

# UPPER COLUMBIA RIVER

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## **Draft Final Quality Assurance Project Plan for the Phase 2 Sediment Study Version 3**

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February 2013



## SECTION A: PROJECT MANAGEMENT

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### A1 TITLE AND APPROVAL SHEET

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#### QUALITY ASSURANCE PROJECT PLAN FOR THE PHASE 2 SEDIMENT STUDY

EPA Project Coordinator	Laura Buelow	_____	Date	_____
EPA Project Coordinator	Matt Wilkening	_____	Date	_____
TAI Project Coordinator	Marko Adzic	_____	Date	_____
EPA Quality Assurance Manager	Ginna Grepo-Grove	_____	Date	_____
TAI Technical Coordinator	Anne Fairbrother	_____	Date	_____
Co-Principal Investigator	Robert Santore	_____	Date	_____
Co-Principal Investigator	Paul Paquin	_____	Date	_____
Senior Technical Advisor	Rick Cardwell	_____	Date	_____
Senior Technical Advisor	Scott Becker	_____	Date	_____
Task QA Manager	Rock Vitale	_____	Date	_____
Analytical Chemistry Laboratory Coordinator	Kris McCaig	_____	Date	_____
Bioassay Laboratory Coordinator	Ashley Kaiser	_____	Date	_____
Chemistry Laboratory Project Manager	Lynda Huckestein	_____	Date	_____
Chemistry Laboratory QA Manager	Suzanne LeMay	_____	Date	_____
Bioassay Laboratory Project Manager	Jeffrey Cotsifas	_____	Date	_____
Bioassay Laboratory QA Manager	Stephen L. Clark	_____	Date	_____
Database Administrator	Randy O'Boyle	_____	Date	_____





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## ACRONYMS AND ABBREVIATIONS

Agreement	June 2, 2006, Settlement Agreement
ACG	analytical concentration goal
AFDW	ash-free dry weight
AIC	Akaike's information criterion
ASTM	American Society for Testing and Materials
AVS	acid volatile sulfide
BERA	Baseline Ecological Risk Assessment
BLM	biotic ligand model
CAS	Columbia Analytical Services
COC	chain-of-custody
COPC	chemical of potential concern
CSM	conceptual site model
DOC	dissolved organic carbon
DQI	data quality indicator
DQO	data quality objective
DMP	data management plan
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
ESB	equilibrium-partitioning sediment benchmark
ESI	Environmental Services, Inc.
Exponent	Exponent, Inc.
foc	fraction of organic carbon
FSP	field sampling plan
GIS	geographic information system
IWTU	interstitial water toxicity unit
LCS	laboratory control sample
LCSD	LCS duplicate
MDL	method detection limit
mPECQ	mean probable effects concentration quotient
MQO	measurement quality objective
MRL	method reporting limit

MS	matrix spike
MSD	matrix spike duplicate
NAWQC	National ambient water quality criteria
NELAC	National Environmental Laboratory Accreditation Conference
NIST	National Institute of Standards and Technology
PARCC	precision, accuracy or bias, representativeness, completeness and comparability
pwBLM	BLM calculation based on sediment porewater composition
QA	quality assurance
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
QC	quality control
RI/FS	remedial investigation and feasibility study
RM	river mile
RPD	relative percent difference
RSD	relative standard deviation
SEM – AVS	simultaneously extracted metals minus acid volatile sulfide (which is defined as ‘excess SEM’. See also “SEMx”.)
SEM	simultaneously extracted metals
SEM <sub>x</sub>	excess SEM; the difference of SEM minus AVS
SEM <sub>x,OC</sub>	carbon normalized excess SEM
SHSP	Site Health and Safety Plan
Site	Upper Columbia River site
SLERA	Screening Level Ecological Risk Assessment
SOP	standard operating procedure
TAI	Teck American Incorporated
TAL	target analyte list
TIE	Toxicity Identification Evaluation
TOC	total organic carbon
UCR	Upper Columbia River
Zn/V	zinc-to-vanadium ratio

## UNITS OF MEASURE

°C	degree(s) Celsius
cm	centimeter(s)
d	day
dw	dry weight
in.	inch(es)
h	hour(s)
km	kilometer(s)
lux	unit of illumination
L:D	light to dark ratio (photoperiod)
m	meter(s)
m <sup>2</sup>	square meter(s)
mg/L	milligram(s) per liter
mL	milliliter(s)
mm	millimeter(s)
µm	micrometer(s)
µS/cm	microSiemens/centimeter
µg/L	microgram(s) per liter
µmol/g	micromoles per gram
µmol/g <sub>d</sub>	micromoles per gram (dry weight)
µmol/g <sub>oc</sub>	micromoles per gram organic carbon
v/v	volume to volume
wwt/wwt	wet weight to wet weight



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### **A3      DISTRIBUTION LIST**

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EPA QA Manager	Ginna Grepo-Grove
TAI Project Coordinator	Marko Adzic
TAI Technical Team Coordinator	Anne Fairbrother
Co-principal Investigator	Paul Paquin
Co-principal Investigator	Robert Santore
Task QA Manager	Rock Vitale
Senior Technical Advisor	Rick Cardwell
Senior Technical Advisor	Scott Becker
Field Supervisor	Dave Enos
Database Administrator	Randy O'Boyle
Analytical Chemistry Laboratory Coordinator	Kris McCaig
Bioassay Laboratory Coordinator	Ashley Kaiser
Chemistry Laboratory Project Manager	Lynda Huckestein
Chemistry Laboratory QA Manager	Suzanne LeMay
Bioassay Laboratory Project Manager	Jeffrey Cotsifas
Bioassay Laboratory QA Manager	Stephen L. Clark





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## **A4 INTRODUCTION AND TASK ORGANIZATION**

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### **A4.1 Introduction**

This document presents the quality assurance project plan (QAPP) for the Phase 2 sediment study (herein the 'study') of the Upper Columbia River (UCR) (herein the 'Site'), which extends from the U.S.-Canada border (river mile [RM] 745) to Grand Coulee Dam (RM 596). This study is one of many tasks being completed as part of the Site remedial investigation and feasibility study (RI/FS) by Teck American Incorporated (TAI) under U.S. Environmental Protection Agency (EPA) oversight. The objective of the RI/FS is to investigate the nature and extent of unacceptable risk at the Site to people and the environment.

This QAPP describes the organization, data quality objectives (DQOs), study design, analytical procedures, and quality assurance and quality control (QA/QC) procedures upon which the study will be based. The field sampling plan (FSP) describes field procedures and protocols that will be followed and is presented in Appendix A.

The primary objective of this study is to evaluate if there are unacceptable risks to benthic invertebrates (herein 'benthos') associated with exposure to metals and other chemicals in UCR sediments. To achieve this, site-specific relationships between chemical of potential concern (COPC) concentrations (including factors affecting bioavailability) and toxicity will be evaluated. Data collection efforts will focus on obtaining information that will inform our understanding of potential relationships between sediment chemistry and toxicity. In addition, data collected during this study will be used to inform other components of the ecological risk assessment (e.g., evaluation of risk to aquatic plants, sediment-probing birds, and other receptors).

EPA's DQO process (USEPA 2006a) was used to guide the development of the requirements and design rationale for data collection activities presented in this QAPP.

### **A4.2 Task Organization**

This section presents the organizational structure for activities associated with the work, including task management and oversight, fieldwork, sample analysis, and data management. Contact information for team task members is provided in Table A4-1.

#### **A4.2.1 EPA Organization and Responsibilities**

EPA will oversee TAI activities associated with the study and will coordinate U.S. Department of the Interior, Washington State Department of Ecology (Ecology), and

tribal (i.e., the Confederated Tribes of the Colville Reservation and the Spokane Tribe of Indians) input with respect to review of technical documents submitted by TAI. In addition EPA, under Section 106 of the National Historic Preservation Act, has the primary responsibility for consulting with interested parties. EPA's project coordinators, Dr. Laura Buelow and Matt Wilkening, will be responsible for ensuring that the work performed is consistent with all applicable EPA guidance. The EPA quality assurance (QA) manager is Ginna Grepo-Grove, or designee.

#### **A4.2.2 TAI Organization and Responsibilities**

Marko Adzic will serve as TAI's project coordinator and will have the primary responsibility for ensuring that TAI meets all the requirements and associated deliverables specified within the June 2, 2006, Settlement Agreement (Agreement) (USEPA 2006b). Dr. Anne Fairbrother will be responsible for overseeing technical aspects of this task.

#### **A4.2.3 Key Task Personnel**

TAI technical team members for the study and their respective responsibilities are identified below.

**Technical Team Coordinator**—Dr. Fairbrother (Exponent, Inc. [Exponent]) will oversee task activities, review QA reports, and ensure that required activities are completed in sequence. Dr. Fairbrother will work closely with the co-principal investigator(s) and task QA coordinator to ensure that all requirements are met and study objectives achieved.

**Co-principal Investigator(s)**—Robert Santore and Paul Paquin (both of HDR | HydroQual Inc.)—will serve as co-principal investigators and will oversee and approve all project activities, review QA reports, approve final project QA needs, and authorize necessary actions and adjustments needed to accomplish program QA objectives. They will provide on-site supervision as needed and ensure that proper sample collection, preservation, storage, transport, and chain-of-custody (COC) procedures are followed. They will inform the technical team coordinator when problems occur and will communicate and document corrective actions taken.

**Senior Technical Advisor(s)**—Drs. Rick Cardwell (Cardwell Consulting, LLC) and Scott Becker (Integral Consulting Inc.) will serve as senior technical advisors for the study, and are responsible for providing technical oversight in the design, implementation, and data interpretation.

**Task QA Coordinator**—Rock Vitale (Environmental Services, Inc. [ESI]) is the task QA coordinator and is responsible for providing overall QA support for the study. Mr. Vitale will coordinate the validation of laboratory data; communicate data quality issues; and work with the database administrator to address potential data limitations. Mr. Vitale will report directly to the analytical chemistry laboratory coordinator, and will work closely with the bioassay laboratory coordinator, the database administrator, and the laboratories to ensure that the data are of the highest quality.

**Bioassay Laboratory Coordinator**—Ashley Kaiser (Exponent) is the bioassay laboratory coordinator and is responsible for ensuring that bioassay method development is completed satisfactorily; coordinating receipt of samples by the test laboratory and tracking the laboratory's progress; addressing QA issues related to the bioassays; and addressing any scheduling issues. Ms. Kaiser will report to the technical team coordinator, and will work closely with the task QA coordinator and the database administrator.

**Analytical Chemistry Laboratory Coordinator**—Kris McCaig (TAI) is the analytical chemistry laboratory coordinator and is responsible for ensuring that laboratory method selection and/or development is satisfactorily completed prior to the analysis of samples; coordinating with the testing laboratory and tracking the laboratory's progress; verifying that the laboratory has implemented the requirements of this QAPP; addressing QA issues related to the laboratory analyses; ensuring that laboratory capacity is sufficient to undertake the required analyses in a timely manner; and addressing scheduling issues related to laboratory analyses. Ms. McCaig will report directly to the TAI project coordinator and will work closely with the technical team coordinator.

**Database Administrator**—Mr. Randy O'Boyle (Exponent) is the database administrator and will have primary responsibility for data management and database maintenance and development. Mr. O'Boyle will be responsible for overseeing and/or conducting the following activities: establishing storage formats and procedures appropriate for data collected; ensuring all data packages are complete and delivered in the correct format; maintaining the integrity and completeness of the database; and providing data summaries to data users for interpretation and reporting. Mr. O'Boyle will report directly to the technical team coordinator and will work closely with the task QA coordinator and laboratories.

#### **A4.2.4 Laboratories**

The following responsibilities apply to respective project and QA managers at the analytical and bioassay laboratories. The analytical laboratory will be Columbia Analytical Services (CAS) while the bioassay laboratory will be Pacific EcoRisk (pending approval by EPA). Each will have the following staff available for this project.

**Analytical Chemistry Laboratory Project Manager**—Lynda Huckestein (CAS) is responsible for the successful and timely completion of sample analyses, as well as the following:

- Ensuring that samples are received and logged correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Reviewing analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs)
- Apprising the laboratory coordinator of the schedule and status of sample analyses and data package preparation
- Notifying the laboratory coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Taking appropriate corrective action as necessary
- Reporting data and supporting QA information as specified in this QAPP
- Providing electronic data deliverables (EDDs) in a format consistent and compatible with the database.

**Analytical Chemistry Laboratory QA Manager**—Suzanne LeMay (CAS) is responsible for overseeing QA activities in the laboratory and ensuring the quality of the data for this task. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program
- Maintain QA records for each laboratory production unit
- Ensure that QA/QC procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve non-conformity and corrective action reports
- Coordinate responses to any quality control (QC) issues that affect this task with the analytical chemistry laboratory project manager.

Roles and responsibilities outlined above for CAS will also apply to Pacific EcoRisk, where Jeffrey Cotsifas and Stephen Clark will serve as the project manager and QA manager, respectively.

## **A5 PROBLEM DEFINITION AND BACKGROUND**

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The Baseline Ecological Risk Assessment (BERA) work plan (TAI 2011) identified several historical studies that collected and evaluated sediment chemistry and toxicity data from the Site. Detailed summaries and an integration of these data are presented within Appendices D (sediment chemistry) and E (sediment toxicity) of the BERA work plan. Similarly, the Screening Level Ecological Risk Assessment (SLERA; TAI 2010) identified a number of COPCs within sediments and associated porewater for which data collected to date are either 1) insufficient to assess the potential for adverse ecological effects, or 2) indicate a potential for adverse ecological effects. As a result, additional data collection and analyses are needed to evaluate potential risks to benthos associated with these COPCs. A summary of sediment/porewater COPCs requiring additional evaluation as determined by the SLERA are presented in Table A5-1. At the direction of EPA, COPCs to be evaluated for this study are limited to target analyte list metals<sup>1</sup>.

Further evaluation of potential risks to benthos using multiple lines of evidence such as sediment and porewater chemistry, as well as whole-sediment toxicity tests, is required. To guide these efforts and ensure that representative areas spanning a range of potential exposures are evaluated, site-specific data were used to examine spatial gradients and define characteristic ranges (i.e., “bins”) for representative sediment bed properties. Specifically, sediment bed properties identified in consultation with EPA were selected to represent a spectrum of site conditions and exposure gradients. These bed properties include zinc-to-vanadium ratio (Zn/V), total organic carbon (TOC), mean probable effects concentration quotient (mPECQ), and sediment texture.

Using geostatistical methods, the aforementioned sediment bed properties were mapped on a continuous basis over the Site. Joint variations of bed properties were used to define groups of sediment that, in turn, were categorized into high, medium, and low

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<sup>1</sup> At the time of writing, a Site-wide COPC refinement document is being prepared and will be submitted under separate cover. This refinement will include and evaluate any and all EPA-approved data as collected for the RI/FS (e.g., beach sediments, surface water, fish tissue etc...); and will refine the assumptions and methods used in the 2010 Screening Level Ecological Risk Assessment. It is not anticipated that results of the aforementioned refinement will adversely affect data collection efforts for this Study as it has been developed to incorporate a tiered-approach (e.g., Toxicity Identification and Evaluation).

exposure gradient bins. A summary of the results (i.e., spatial distribution of sediment groups) is detailed in Appendix B<sup>2</sup>.

## **A6 DATA NEEDS**

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Independent studies conducted to date at the Site have identified a number of sediment COPCs that may adversely affect benthos. These studies do not, however, sufficiently establish potential concentration-response relationships, nor do they fully integrate measures of bioavailability (USEPA 2007). As a result, the primary purpose of this study is to evaluate potential risks to benthos associated with exposure to sediment/porewater COPCs<sup>3</sup>. To do this, additional sediment/porewater chemistry data and synoptic benthic toxicity tests are needed. In addition, sediment and porewater data collected during this study can and will be used to inform other components of the BERA. For example, these data can and will be used, as appropriate and applicable, in the evaluation of unacceptable risks to other ecological receptors such as aquatic plants and sediment-probing birds, see Figure A6-1. Furthermore and if required by EPA, invertebrate tissue chemistry from *Hyalella azteca* collected after Toxicity Identification Evaluation (TIE) testing will be considered as a secondary line of evidence.

## **A7 DATA QUALITY OBJECTIVES, CRITERIA, AND DESIGN RATIONALE**

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EPA's seven-step DQO process (USEPA 2006a) was used to guide the design rationale for the Phase 2 sediment study. Each step is described below.

### **A7.1 Step 1—State the Problem**

As noted in Section A6, studies conducted to date have identified a number of sediment COPCs that may adversely affect benthos within the UCR. These studies do not

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<sup>2</sup> As documented on July 3, 2012, on the basis that sampling can proceed (see Section A7.1.2 herein), TAI while reserving its right to raise technical concerns associated with EPA's alternate locations (refer to June 11, 2012 correspondence), will undertake sediment sampling activities and analyses at EPA's alternate locations (refer to April 27, 2012 letter to TAI). TAI, also under protest, has incorporated the site reconnaissance recommendations outlined by EPA's contractor (CH2M Hill, Inc.; June 27, 2012 technical memorandum). As a result, although the methods presented herein (including the Appendix) may not have fully been considered for EPA's program, they remain appropriate. In addition and as requested by EPA, materials presented within Appendix B, may be updated following data collection and the analyses outlined herein.

<sup>3</sup> The primary purpose is consistent with EPA's February 2010 level-of-effort paper, which states "the goal of this sediment sampling component of the baseline ecological risk assessment (BERA) is to evaluate risks to benthic invertebrates associated with exposure to metals and other chemicals in the UCR [as identified by the screening-level ecological risk assessment (SLERA) for the site]."

sufficiently establish potential concentration-response relationships, nor do they fully integrate measures of bioavailability (USEPA 2007). Accordingly, this study will characterize factors that influence bioavailability of COPCs in sediment and assess if unacceptable risks to benthos exist. Application of concentration-response relationships to benthic bioassay data and associated chemistry (sediment/laboratory and field porewater) will provide a basis for evaluating potential risks to benthos throughout the UCR. Sediment and field porewater data collected during this study can and will also be used to inform other components of the BERA (e.g., in the evaluation of risk to aquatic plants, sediment-probing birds, and other receptors). Furthermore, sediment/field porewater data collected during this study will be used to refine spatial gradients; sediment bed properties such as slag content (e.g., Zn/V ratio<sup>4</sup>), TOC, mPECQ, and sediment texture (refine the nature and extent of unacceptable risk at the Site<sup>5</sup>).

#### **A7.1.1 Team Members and Roles**

Team members and their roles are described in Section A4.2 of this QAPP.

#### **A7.1.2 Schedule**

It is anticipated that this work will be completed in early to mid-fall (September to October). For planning purposes, it is anticipated that preliminary results will be available by late winter (December). These preliminary data will be used to help guide, inform, and refine which samples will undergo additional long-term toxicity tests and specialized analyses such as backscatter electron microscopy. It is acknowledged that prior to initiating the aforementioned additional tests and specialized analyses, technical memoranda, or amendment(s) to this QAPP will be required. As a result, the above-mentioned schedule is for planning purposes only and is subject to change.

Following Phase 2 sediment/toxicity data collection, analyses, and evaluation, if the EPA determines that there is insufficient information to support an informed risk-based management decision using existing site data; additional sediment/toxicity sample collection may be needed. The need for future sampling will be data driven and directed by EPA, if determined to be necessary.

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<sup>4</sup> The basis and rationale of using a Zn/V ratio was detailed within Appendix D of the BERA work plan (TAI 2011). Other chemical ratios and/or methods (i.e., backscatter electron microscopy) may also be used to refine sediment bed properties and facilitate data interpretation.

<sup>5</sup> The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

## **A7.2 Step 2—Identify the Goal of the Study**

Consistent with EPA's February 2010 level-of-effort, this study was "designed to evaluate the risks to benthic invertebrate communities." As a result, the primary goal of this study is to evaluate risks to benthos associated with exposure to COPCs in UCR sediments. Specific DQOs to be addressed are as follows:

- Are sediment COPCs bioavailable at levels indicative of potential unacceptable risks to benthos?
- Are there significant differences in survival, growth, or reproduction of benthos (i.e., amphipods and midge) exposed to Site and reference sediments? If significant differences occur
  - What is the magnitude of these effects?
  - Are these effects due to COPCs as measured in sediments and/or porewater?
  - What concentration-response relationships can be established between measured COPC concentrations and observed effects?

In addition to the above-mentioned primary goal and associated DQOs, other questions to be addressed by this study include

- Are sediment COPCs bioavailable at levels indicative of potential unacceptable risks to other ecological receptors (e.g., aquatic plants, sediment-probing birds)?
- Can the nature and extent of unacceptable risk at the Site via spatial gradients and sediment bed properties such as slag content (e.g., Zn/V ratio<sup>6</sup>), TOC, mPECQ, and sediment texture be further refined?<sup>7</sup>

## **A7.3 Step 3—Identify Information Inputs**

The third step of the DQO process (USEPA 2006a) requires consideration of the following:

- Types and potential sources of information (e.g., site characteristics or variables) that should be measured to provide estimates or resolve decisions
- Information to provide a basis for specifying performance or acceptance criteria
- Information on the performance of appropriate sampling and analyses methods.

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<sup>6</sup> The basis and rationale of using a Zn/V ratio was detailed within Appendix D of the BERA work plan (TAI 2011). Other chemical ratios and/or methods (i.e., backscatter electron microscopy) may also be used to refine sediment bed properties and facilitate data interpretation.

<sup>7</sup> The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.



Determination or estimation of unacceptable risks to benthos and other ecological receptors (refer to Figure A6-1) requires representative data on bioavailability for COPCs in Site sediments as collected over a range of exposure gradients. Samples collected along anticipated exposure gradients will facilitate the collection of representative Site sediments, and the evaluation of potential concentration-response relationships and unacceptable risks. The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

The degree of COPC bioavailability will be measured and evaluated using a range of methods. These include, but are not limited to, mPECQ, excess simultaneously extracted metals (SEM<sub>x</sub>) (simultaneously extracted metals minus acid volatile sulfide [SEM – AVS]), carbon-normalized excess SEM (SEM<sub>x,oc</sub> = SEM<sub>x</sub>/fraction organic carbon), pH, and the biotic ligand model (BLM). Significant differences in the survival, growth, or reproduction of benthos will be evaluated using synoptic whole-sediment bioassays with standard test organisms (i.e., amphipods and midge) and sediments collected at the Site and in one or more reference areas.

The adequacy of multiple metal ratio methods for describing sediment bed properties such as slag content will be evaluated by using field observations (e.g., presence/absence and percent of visible black silica glass particles) in conjunction with sediment chemistry. Sediment samples will be archived and no fewer than 35 samples will undergo backscatter electron microscopy following a review of the preliminary data. Samples will be selected for this specialized work following a review of the preliminary chemistry data; and will be documented in a technical memorandum, or QAPP addendum, for EPA's review and approval.

Information from both field and laboratory chemistry (sediment/porewater) and bioassay endpoints will be used to identify areas of unacceptable risk to benthos and evaluate concentration-response relationships.

### **A7.3.1 Sediment and Field Porewater Chemistry**

Whole sediment and field porewater chemistry will be collected from 140 sampling stations. This total includes 124 Site samples (10 of which are intended to be internal references), and 16 external reference samples (Table A7-1). External reference samples include 6 tributary reference, and 10 upstream reference samples. Samples will be collected and analyzed from the top 6 in. (15 cm) of the sediment (i.e., the depth commonly associated with the biologically active zone). To evaluate the degree to which sediment COPCs may be bioavailable and indicative of potential unacceptable risks, the following analytical measurements will be conducted on all samples.

### Whole-Sediment Chemistry

Sediment samples will be analyzed for grain size, pH, AVS (acid volatile sulfide), SEM, TOC, and target analyte list (TAL) metals<sup>8</sup>. EPA methods for analyses of bulk sediment chemistry are listed in Table A7-2.

### Field Porewater Chemistry

Field porewater samples will be collected *ex situ* via suction (i.e., airstones). In short, this will involve the careful insertion (horizontally) of an airstone within the sediment as it remains in the sampling equipment (i.e., Van Veen sampler) at the time of sample collection (prior to any compositing that may be performed). Upon insertion, the top of the airstone will sit approximately 3 in. (7 cm) below the sediment surface. The airstone will be connected to a large ( $\leq 140$  mL) syringe via decontaminated polyethylene tubing through which field porewater will be extracted.

If sufficient volume is available, field porewater samples will be analyzed for TAL metals (the dissolved fraction) and other water quality parameters needed to assess metal bioavailability using the BLM. Therefore, the volume-dependent priority order of porewater analytes includes 1) aluminum, cadmium, calcium, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, and zinc; 2) pH, dissolved organic carbon [DOC], hardness (to be calculated), and alkalinity; and 3) chloride and sulfate. Chemical analyses will be performed according to EPA methods (Table A7-2).

### A7.3.2 Whole-Sediment Bioassays

Of the 140 sampling stations identified and discussed in Section A7.3.1, whole-sediment bioassays using the amphipod *Hyalella azteca* and the midge *Chironomus dilutus* will be synoptically performed on 74 (53 percent) of the samples, in accordance with EPA<sup>9</sup>; refer to Maps A7-1 through A7-9. Specifically, bioassays to be performed on all 74 samples include the following:

- 28-day whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of survival, weight, and biomass [USEPA 2000; ASTM 2012])
- 10-day whole-sediment toxicity tests with the midge, *C. dilutus* (endpoint of survival, weight, and biomass [USEPA 2000; ASTM 2012]).

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<sup>8</sup> TAL metals include aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.

<sup>9</sup> The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

In addition to the above-listed standard bioassays, reproductive endpoints will be also assessed on 18 split-samples. Consistent with EPA's direction, preference for these 18 split-samples will be given to those stations located within high and medium exposure gradient bins, but will be finalized following review of preliminary data. Results of the above-listed 10- and 28-day survival and growth tests, in conjunction with preliminary chemistry data will be used to refine and identify which samples will undergo further evaluation; and will be documented in a technical memorandum, or QAPP addendum, for EPA's review and approval. Specific bioassays to be performed on these 18 samples include the following:

- 42-day whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of survival, weight, biomass, and neonates/surviving female [USEPA 2000; ASTM 2012])
- 50- to 65-day whole-sediment toxicity tests with the midge, *C. dilutus* (endpoints of survival, weight, biomass, emergence, eggs/surviving female, egg hatching, and viability of young using <24 hour old larvae [USEPA 2000; ASTM 2012]).

To meet Study DQOs and minimize the potential for confounding inter-batch variability with other variables (e.g., due to a chemical gradient), short-term bioassay testing will be initiated only after completing all field sampling. Short-term bioassays will be conducted in multiple batches, with each batch consisting of up to approximately 15 samples plus controls. Samples will be assigned to batches using a stratified random approach. The strata will be based on river reaches to ensure that each batch will contain samples from across all geographic areas of the river (including external reference locations), to the maximum extent possible. Within strata, samples will be randomly selected for each batch. Upon identifying and assigning bioassay samples in respective batches, the stratified random bioassay batching scheme will be reviewed and approved by EPA prior to bioassay testing.

Bioassay results will be used to evaluate if the survival, growth, or reproduction of benthos in Site sediments differ significantly from those in reference sediments. One approach that will be used to conduct this analysis is application of the "reference envelope" approach which examines whether responses from Site samples lie below the range of results from reference samples (Hunt et al. 2001). If significant differences are identified, these data will also aid to evaluate and address a) the magnitude of these effects; and b) a concentration-response relationship between COPCs and observed effects. These results will also be used to evaluate the relative value of respective bioassays for other potential sampling efforts.

Should equivocal or unexplained responses be identified in the bioassays, further evaluation (i.e., TIE) will be completed if required to discern the class of chemicals resulting in the observed effects, or if these responses are a consequence of non-chemical properties. TIEs would be conducted according to EPA guidance and studies reported in the scientific literature (e.g., Ho et al. 2007; Hockett and Mount 1996); see Section B4.2.2 of this QAPP for further details. Associated with TIE testing and if required by EPA, invertebrate tissue chemistry from *H. azteca* collected after TIE testing will be considered as a secondary line of evidence.

In addition to the aforementioned bioassays, no fewer than 35 sediment samples will be selected for backscatter electron microscopy. Preliminary results (e.g., chemistry data, field observations etc.) will be used to refine and identify which samples will undergo this evaluation. Samples to be tested, the detailed approach, and associated QA/QC requirements will be documented in a technical memorandum for EPA's review and agreement.

#### **A7.4 Step 4—Define the Boundaries of the Study**

This step specifies the population of interest for the study, the geographical boundaries of the Site, and any temporal considerations that may be required.

##### **A7.4.1 Target Populations for Risk Evaluation**

Target populations of primary interest are benthos that live in or on UCR sediment; and other ecological receptors (e.g., sediment-probing birds) as identified within the conceptual site model, refer to Figure A6-1. *H. azteca* and *C. dilutus* consistently have demonstrated to be sensitive indicator organisms for sediment contamination, particularly for metals (Milani et al. 2003); therefore, they are protective of target populations of interest. Consistent with Guidance (USEPA 1997), should EPA determine that there is insufficient information to support an informed risk-based management decision using existing site data (includes data from this study), additional sediment/toxicity data may be needed. Such studies may include the use of other test organisms (e.g., freshwater mussels) should information within the scientific community indicate they are better suited to evaluate sediment contamination, and if standard test methods approved by American Society for Testing and Materials (ASTM) or EPA are available.

##### **A7.4.2 Geographic Boundaries of the Site**

The Site, as stated in Section A4.1 of this document, encompasses the UCR from the U.S.-Canada border (RM 745) to the Grand Coulee Dam (approximately RM 596).

Sediments will be collected and analyzed (chemically and toxicologically) from representative locations throughout the UCR. Reference sediments will be collected from locations upstream (north) of the Site and those identified by EPA (April 27, 2012 correspondence), see Maps A7-7 through A7-9.

#### **A7.4.3 Temporal Considerations**

Samples will be collected in the fall of 2013 from representative areas throughout the UCR and will be used to refine exposure gradients, identify areas of potential unacceptable risk to benthos, and evaluate relative responsiveness of bioassay endpoints. Preliminary data will be used to guide, inform, and select samples which will be analyzed for reproductive endpoints, backscatter electron microscopy, and TIE investigations (if necessary). Consistent with EPA Guidance (USEPA 1997), should EPA determine that there is insufficient information to support an informed risk-based management decision using the above-mentioned data in association with other existing site data (e.g., Phase 1 sediment/toxicity data; [USEPA 2006c]); additional sediment/toxicity data may be needed. Furthermore and per the terms and conditions of the Agreement, should TAI identify the need for additional data; this would be documented in a technical memorandum.

#### **A7.5 Step 5—Define the Statistics and Types of Inferences**

Step 5 of the DQO process provides data analysis approaches that will be used to evaluate the data and draw conclusions on risks to benthic receptors and other ecological receptors. It is necessary to have a general understanding of the types of data analyses that will be conducted to ensure that the required parameters are measured, and that a sufficiently large data set is developed to provide the desired level of confidence in the statistics. This approach will ensure the generation of a data set that will be adequate for use in conducting the baseline ecological risk assessment.

This section briefly describes how bioavailability parameters will be incorporated into the analysis to determine toxicity of sediments. Statistical methods for determining which bioassays are toxic are described as well as how concentration-response relationships between bioavailable concentrations of COPCs in sediment or porewater and toxic effects on benthos will be derived.

##### **A7.5.1 Estimation of Bioavailability**

Consistent with EPA's suggestion<sup>10</sup>, the lines of evidence and the refinement of sediment bed properties (refer to Appendix B) may be updated and refined using sediment and

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<sup>10</sup> Refer to specific comment number four from EPA's June 21, 2012 correspondence to TAI.

porewater data collected from this and other site-specific data (i.e., beach sediment and white sturgeon sediment toxicity data). Such analyses may aid in evaluating the nature and extent of unacceptable risk within the Site<sup>11</sup>. Because environmental factors can alter the bioavailability of contaminants these bioavailability effects can confound relationships between organism response and the total (bulk) concentration in sediments. Therefore, it is likely that a stronger relationship (e.g., correlation) between sediment characteristics and bioassay responses will be evident once data are adjusted to account for site-specific bioavailability. Preparation of samples for laboratory bioassays necessarily results in changes to sediment characteristics that affect bioavailability, such as amount of AVS present (dependent upon degree of oxidation of the sediments), the chemistry of sediment porewaters, and particle size. Therefore, the analyses described in Section A7-3 will be performed not only with synoptic chemistry and bioassay data, but also with chemistry-only samples (sediment and porewater measurements).

Observations and data identifying sediments where metals are most likely to pose unacceptable risks to benthos, will be supported by an analysis of the relationship of positive bioassay responses with concentrations of AVS, SEM, TOC, and other important constituents that affect bioavailability (i.e., other binding ligands and competing cations). If positive responses are seen when they are predicted to not occur (e.g., in sediment samples with high AVS and/or organic carbon), this will provide a line of evidence that metals are not causing the positive response seen in the bioassay. Other lines of evidence, such as the TIE or concentrations of organic chemicals, will then need to be examined to see if they are better at explaining the observed responses. When used in conjunction with bioassay data, excess SEM and carbon normalized excess SEM is expected to improve the statistical quality of the data, and lead to a more thorough understanding of the causes of observed toxicity.

Because excess SEM tends to be a conservative approach (it can identify sediments that are not toxic, but is not very good at identifying those with moderate toxicity; refer to USEPA [2007]), a second line of evidence using porewater chemistry will be employed. One such approach entails the application of interstitial water toxic units (USEPA 2007) for the SEM metals, another relatively conservative assessment method. In addition, we will consider the results of an application of the BLM to porewater collected in both the laboratory (bioassays) and the field to determine site-specific toxicity thresholds.

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<sup>11</sup> The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

#### **A7.5.2 Analysis of Bioassay Data**

EPA guidance will be followed concerning statistical analysis of sediment toxicity data when analyzing results from the whole-sediment bioassays (USEPA 2000). As such, a variety of methods will be used to evaluate these data. Samples that exhibit adverse responses relative to reference samples will be further evaluated to determine if the responses are related to COPCs. Additional detail regarding the consideration and selection of reference sites is discussed in Section B1.1 of this document. A reference envelope approach (e.g., Hunt et al. 2001) will also be applied to the data, where reference site responses will be used to develop a response distribution and select a lower tolerance limit (e.g., generally the 5th percentile) to evaluate Site responses. Site samples with responses (e.g., survival or biomass) below the tolerance limit would be considered a “positive” response.

Samples exhibiting positive bioassay responses could be further analyzed through a TIE (USEPA 2007; Ho et al. 2007; Hockett and Mount 1996) to determine the likely causative factor(s) of the toxic response. Simply, the TIE methodology involves the physical and chemical manipulation of the sample to methodically alter the potency of different chemical classes. Biological responses are then used to gauge the relative change in toxicity caused by these manipulations. Three types of manipulations of bulk sediment samples could be implemented 1) cation exchange resin or sulfide addition to sequester and reduce the toxicity of metals; 2) coconut charcoal or Ambersorb® addition to sequester and reduce the toxicity of organic compounds; and/or 3) Zeolite addition to reduce ammonia toxicity.

If any of the aforementioned manipulations are demonstrated to result in a significant reduction in toxicity versus that of non-manipulated sediments, this would suggest that the targeted chemical class is the primary driver of the positive response. Subsequent phases of the TIE process could be implemented to pinpoint specific COPCs as causative factors of the sample toxicity, if deemed necessary.

#### **A7.5.3 Concentration-Response Relationships**

Exploratory data analysis will be conducted to determine which, if any, measured parameters are most correlated with observed toxicity responses. Data generated in this study will be sufficient to support a variety of statistical analyses, including but not limited to regression analyses (e.g., stepwise linear regression) or the more parsimonious method used in information theoretic approaches (e.g., Akaike's information criterion [AIC]). Principal component analyses also might provide information about which group of analytes are most likely associated with positive bioassay responses, although these analyses will not provide a quantitative relationship. Note that these analyses will

be conducted based on bulk sediment and laboratory porewater data. This type of data analysis will provide one line of evidence, but because it is based on correlative parameters it is not a very good predictor of causality. For example, if an analyte that is not causing toxicity changes its concentration in the same relative amount as a physical stressor (e.g., particle size), then it may appear that the analyte is the cause of the response when in reality it is not. Nevertheless, such correlative relationships may be helpful in site management once causality is more definitively established.

The adequacy of multiple metal ratio methods for describing sediment bed properties such as slag content will be evaluated by using field observations (e.g., presence/absence and percent of visible black silica glass particles) in conjunction with sediment chemistry (e.g., aluminum, calcium, copper, iron, vanadium, and zinc). This analysis will facilitate the identification and selection of select samples for backscatter electron microscopy. Sample selection for this specialized work will be documented in a QAPP addendum for EPA's review and approval.

## **A7.6 Step 6—Specify Performance or Acceptance Criteria**

The goal of Step 6 is to define performance or acceptance criteria to minimize the possibility of either making erroneous conclusions or failing to keep uncertainty in estimates to within acceptable levels (USEPA 2006d). For this study, performance and acceptance criteria will apply to generating appropriate and acceptable data for use during risk assessment activities, as well as providing sufficient data to reduce uncertainty and the probability for false positive or false negative decision errors<sup>12</sup>.

### **A7.6.1 Sampling Completeness**

As demonstrated by previous sampling experience at the site (e.g., USEPA 2006e), the percentage of successful collection of sediment cannot be determined *a priori* because of the unforeseen challenges at some areas, such as sample refusal due to bedrock and/or large cobbles, (i.e., sediments generally having particle diameters greater than 2 mm). Because a large number of backup stations are available to mitigate such potential challenges, the overall goal is to collect 100 percent of the targeted samples representing each of the sample bins. To move to an alternative location the field sampling team will consult with EPA or their designee as to the benefit of continuing to attempt to collect a

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<sup>12</sup> Because of variability in collected data, statistical analysis can lead to varying decision outcomes. A false negative decision error (Type II), for example, is when examination of the data leads to a conclusion of no risk, when there is a true potential risk, while a false positive decision error (Type I) indicates a potential risk, when the true risk is negligible (USEPA 2006c).



sample at a site where minimal or no appropriately sized sediment is available. Final determination of the study success will be evaluated against the DQOs.

#### **A7.6.2 Data Quality**

Techniques for sediment and field porewater sample collection must provide samples of sufficient volume that are collected from appropriate depths. Inferences about these attributes will be based on field observations and a limited number of analytical measurements of critical parameters (e.g., see recommendations for reference area sediments). Precision will be determined by repeatability of chemical measures in duplicate samples (see below).

DQOs are developed using EPA's DQO process (USEPA 2006a) to describe data and data quality needs. Data quality indicators (DQIs) such as the precision, accuracy or bias, representativeness, completeness, and comparability (PARCC) parameters and analytical sensitivity will be used to assess conformance of data with QC criteria (USEPA 2002a).

Field QC samples will include trip blanks, equipment rinsate blanks, field duplicate samples, and certified reference materials. These QC samples will be collected or prepared by sampling personnel in the field and submitted to the laboratory as natural samples.

Equipment rinsate blanks will be used to identify possible contamination from the sampling environment or from sampling equipment. These blanks will be collected by pouring deionized or distilled water over (or through) decontaminated sampling equipment and into a sample jar. One equipment rinsate blank will be collected for each type of sampling equipment used during the sampling event (at an interval of one per day) and will be analyzed for the previously listed metals.

Field split samples will be collected to assess the homogeneity of sediment samples collected in the field and the precision of the sampling process. Field splits will be prepared by collecting two aliquots of sample from the homogenized sediment and submitting them for analysis as separate samples. Field splits will be prepared from at least 10 percent of the sampling locations.

An experimental blank will be used to identify possible contamination from the laboratory and will be collected according to laboratory protocols. Experimental blanks will be collected once per sampling event.

A matrix spike/matrix spike duplicate (MS/MSD) will be performed in the laboratory to assess the accuracy of the analyses. The MS/MSD will be performed according to the laboratory protocols and will occur at a frequency of once every 20 samples.

Method detection limits (MDLs) and method reporting limits (MRLs) for sediment and porewater samples are summarized in Table A7-3, and were selected to ensure consistency with EPA's sediment detection limit evaluation process (USEPA 2008).

Test organism survival should be high prior to the start of the bioassays (e.g., ≥80 percent for 48 hours before the start of a test [USEPA 2000; ASTM 2012]) and survival should remain high (e.g., mean survival of 80 percent for *H. azteca* and 70 percent for *C. dilutus*) in test controls throughout the test duration. Additionally, minimum growth or size requirements may be set for control organisms to ensure that the test population is developing within an acceptable range.

Also, hardness, alkalinity, and ammonia measurements should vary by less than 50 percent over the duration of the exposure, and overlying water-dissolved oxygen concentrations should be maintained at greater than 2.5 mg/L (USEPA 2000).

#### **A7.7 Step 7—Develop the Plan for Collecting Data**

Detailed discussions of the various study components are presented in Section B1 of this QAPP. Because field sampling methods associated with this study involves sediment collection or penetration and disturbance, TAI and its technical team will work with potentially affected parties to assess the effects of the planned work and seek ways to avoid, minimize, or mitigate any adverse effects on historic properties. A cultural resources coordination plan (Appendix C) has been prepared to provide relevant background information about Site-related cultural resources, define measures for protecting resources, and define procedures for consulting with the appropriate state, federal, and tribal parties with interests in the cultural resources of the UCR.

### **A8 SPECIAL TRAINING/CERTIFICATES**

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TAI has assembled a technical team with the requisite experience and technical skills to successfully complete the study. Minimum training and certification requirements for laboratory personnel are provided in the laboratory QA plans (Appendices D and E).

The bioassay laboratory must demonstrate experience with the conduct of all four of the bioassays to be used in this study, as well as the TIE procedure. Accreditation from the National Environmental Laboratory Accreditation Conference (NELAC) is desirable, but not a requirement.

Sampling personnel will be familiar with the Site cultural resources coordination plan (Appendix C). Sampling personnel will report any materials that might be considered a cultural resource to cultural resource observers participating in the field sampling program.

## **A9 DOCUMENTATION AND RECORDS**

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This section identifies on-site and laboratory records to be maintained for this project, information to be included in project reports, data reporting format for data report packages, and document control procedures to be used. Critical records required for this study are identified below with descriptive or supporting information as appropriate. Records will include documents and electronic deliverables related to field sampling (field notebook, sample logs, COC, etc.), toxicity testing, and chemistry laboratory documentation (laboratory records, data packages, project reports, electronic deliverables, etc.), data validation, and data reports. Data reports will be made available through integration into the project web tool. Briefly, this will be an electronic data management system that is accessible via an external web site. The QAPP, FSP (Appendix A), Site Health and Safety Plan (SHSP) (TCAI 2007), and the general SHSP addendum (Attachment A1 to Appendix A) will be provided to each person listed in Section A3. Any revisions or amendments to any of the documents that comprise the FSP will also be provided to these individuals.

### **A9.1 Field Documentation**

The TAI technical team field supervisor will ensure that the field team receives the final approved version of the QAPP prior to the initiation of field activities. Minimum field records that will be maintained include the following:

- Field logbooks
- Photo documentation
- Field data forms
- Sample tracking/COC forms.

Additional content, information, and use of the above-listed documents are further described in the FSP (Appendix A).

## **A9.2 Chemistry Laboratory**

Full laboratory data reports will be provided in electronic format to the task QA coordinator, who will oversee data verification and validation, as well as archiving the final data and data quality reports in the project file. EDDs will be prepared in spreadsheet format and will be compatible with the TAI technical team's database.

Documentation requirements for the analytical laboratory (CAS) are detailed in the QA manual (Appendix D) and will, at a minimum, include the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- Sample receipt and analysis dates
- Final analyte concentration including reporting limit, laboratory qualifiers, and reanalysis
- Percent recovery of each compound in the matrix spike sample
- Matrix spike recovery control limits
- Relative percent difference (RPD) for all MS/MSD and/or laboratory control sample (LCS)/LCS duplicate (LCSD) results
- RPD control limits for MS/MSD and/or LCS/LCSD reports
- LCS results when analyzed
- Recovery control limits for LCS or standard reference material recoveries and relative standard deviation
- Blank results for method blanks, experimental blanks, and equipment blanks
- Method blank summary indicating associated samples
- Case narrative.

## **A9.3 Bioassay Laboratory**

The bioassay laboratory will provide a data package for each sample delivery group or analysis batch that will contain all information required for a complete QA review, including the following:

- A cover letter discussing bioassay procedures and any difficulties encountered
- A case narrative referencing or describing procedures used and any analytical problems and deviations from SOPs and this QAPP
- COC and cooler receipt forms

- A summary of the bioassay results
- Results for all QA/QC checks, including serial dilutions, LCS and reference toxicant tests, and any other QC procedures required by applicable method protocols and laboratory SOPs
- The laboratory toxicity report will document the source of control sediment and associated measurements
- The laboratory toxicity report will document how organisms of known age were obtained for testing
- The weight of a representative subsample of organisms at the start of sediment exposures will be documented
- The laboratory toxicity report will document the measured light intensity during testing
- Original data reports and laboratory worksheets as applicable.

#### **A9.4 Data Quality Documentation**

Data verification (i.e., confirming the accuracy and completeness of field and laboratory data) will be performed by the TAI technical team for data generated in the field, and by each laboratory for the analytical data that it generates. Data validation and data quality assessment for this task will be completed and provided to the task QA coordinator.

Accuracy of the laboratory EDDs will be verified by, or under the direction of, the database administrator. All changes to data stored in the database will be recorded in the database change log. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratories and during data validation.

Data validation reports will be prepared and provided to the laboratory QA manager. Any limitation to the usability of the data will be discussed in this report. Completed data validation checklists will also be provided to the task QA coordinator by the data validator.



## **SECTION B: DATA GENERATION AND ACQUISITION**

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### **B1 SAMPLING PROCESS DESIGN AND RATIONALE**

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This section presents the detailed design and rationale for the sediment study that will result in a data set that supports assessing risk to benthos and other ecological receptors (e.g., sediment-probing birds). The sampling approach was developed based on information from previous investigations and information on sediment COPCs and their potential toxicity<sup>13</sup>.

#### **B1.1 Sampling Locations and Rationale**

Determination or estimation of unacceptable risks to benthos requires representative data on bioavailability of COPCs in sediments as collected over a range of exposure gradients. A summary of the sampling locations, associated rationale, and site reconnaissance is provided in Appendix F.

In addition, to account for uncertainties such as culturally sensitive areas, and/or sediments that cannot be tested due to large grain sizes (e.g., gravels and cobbles) alternative sampling locations have been identified. Refer to Maps A7-1 through A7-6 and Table B1-1 for a summary of proposed and reserve sampling locations and their associated coordinates.

Evaluating sediment toxicity through the use of bioassays requires collection and use of reference sediment samples. EPA (USEPA 1994) has identified a number of desirable characteristics for bioassay reference locations for use in a RI/FS, and for bioassessments of non-wadeable streams (USEPA 2006a). They include the following:

- Upgradient in the same watershed as the study site
- Comparable physical setting as the study site
- Similar water depth and flow as the study site

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<sup>13</sup> As documented on July 3, 2012, on the basis that sampling can proceed (refer to Section A7.1.2 herein), TAI while reserving its right to raise technical concerns associated with EPA's alternate locations (refer to June 11, 2012 correspondence), will undertake sediment sampling activities and analyses at EPA's alternate locations (refer to April 27, 2012 letter to TAI). TAI, also under protest, has incorporated the site reconnaissance recommendations outlined by EPA's contractor (CH2M Hill, Inc.; June 27, 2012 technical memorandum). As a result, although the methods presented herein (including Appendices) may not have not fully been considered for EPA's program, they remain appropriate for this document. In addition and as requested by EPA, materials presented within Appendix B, may be updated following data collection and the analyses outlined herein.

- Similar sediment grain size distribution, sediment TOC content, and water quality as the study site
- Relatively uncontaminated or minimally impaired.

Considering these approaches, the following desirable characteristics and/or performance standards will be considered as part of identifying internal references as well:

- Similar sediment grain size distribution
- Uncontaminated (e.g.,  $mPECQ_{metals} < 0.2$ ; [USEPA 2010])
- Survival and growth will meet the test acceptability criteria for control sediment (USEPA 2000; ASTM 2012).

Given the above-listed desirable characteristics, a preferred reference area is located upstream from the Site (in the same watershed), is relatively uncontaminated, and has similar grain size distribution and TOC content. Therefore, this study will target the acquisition of about 16 external reference locations, of which a minimum of 10 will be located in Canada (i.e., Columbia River at Genelle and Lower Arrow Lake). In addition, and per EPA's letter listing alternate locations<sup>14</sup>, reference areas sampled in 2005 will be resampled for this study.

## B1.2 Bioavailability Measurements

It is important to recognize that the bioavailability of sediment or porewater COPCs is not necessarily a constant fraction of total COPC concentrations but is dependent on the nature of the sediment matrix and concentrations of other constituents (e.g., calcium, magnesium, potassium, sodium, and chloride) affecting chemical speciation and/or biological responses. Sediment conditions or chemical properties that have been integrated into the design to assess COPC bioavailability include the following:

- **AVS and SEM.** EPA (USEPA 2007) recognizes the utility of AVS and SEM for assessing the absence of toxicity of sediments contaminated with selected metals (i.e., silver, cadmium, chromium, copper, lead, nickel, mercury, and zinc) as part of the equilibrium sediment partitioning benchmark (ESB) approach (USEPA 2005). These chemical characteristics are used to define excess SEM ( $SEM_x$ ; the difference  $SEM - AVS$ ) and have utility for identifying locations

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<sup>14</sup> As documented on July 3, 2012, on the basis that sampling can proceed (refer to Section A7.1.2 herein), TAI while reserving its right to raise technical concerns associated with EPA's alternate locations (refer to June 11, 2012 correspondence), will undertake sediment sampling activities and analyses at EPA's alternate locations (refer to April 27, 2012 letter to TAI) which includes the tributary reference locations.



where toxicity to benthos due to SEM metals is not expected. Specifically, when  $AVS \geq 0.1 \mu\text{mol/g}$ , benthos should be adequately protected if SEM does not exceed AVS by more than  $1.7 \mu\text{mol/g}_d$  (i.e.,  $SEM_x = SEM - AVS \leq 1.7$ ). While this approach is predictive of sediments that are not toxic, it can only identify relative risk for sediments when  $SEM_x > 0$ , at which point additional information (e.g., further characterization) is generally recommended (Ankley et al. 1996; USEPA 2005). This is due to the fact that some sediment with  $SEM_x > 0$  may not result in toxicity, because factors other than AVS are modifying the bioavailability of sediment metals (e.g., association of metals with other binding phases such as sediment organic matter or metal oxides). On the basis of  $SEM_x$ , it has been shown that sediments with  $SEM_x < 1.7 \mu\text{mol/g}_d$  pose low risk of adverse biological effects, whereas sediments with  $SEM_x > 120 \mu\text{mol/g}_d$  may be expected to cause adverse biological effects. For  $SEM_x$  between 1.7 and  $120 \mu\text{mol/g}_d$ , the potential for toxicity is uncertain. Because of this uncertainty, if the threshold for no effects is exceeded, then EPA recommends that additional information be considered (USEPA 2005).

$SEM_x$  is useful because metal sulfides are among the most insoluble and tightly bound forms of metals in sediments, and consideration of AVS in the determination of  $SEM_x$  accounts for that portion of the sediment metals that are expected to be bound to sulfide minerals. Other important binding phases in sediments containing  $SEM_x$  may be associated with organic matter.

- **TOC.** Some metals (e.g., copper) and many organic chemicals bind strongly to organic materials in the sediment, thereby altering their potential toxicity. Measurement of TOC can be used to carbon normalize excess SEM ( $SEM_{x,OC} = SEM_x / f_{OC}$ ; where  $f_{OC}$  is the fraction of sediment organic carbon  $\equiv$  TOC (Ankley et al. 1996; USEPA 2005). A refined predictor of toxicity can be achieved when the organic carbon content of the sediment is also considered in the determination of  $SEM_{x,OC}$ .

Sediment with low carbon-normalized  $SEM_x < 130 \mu\text{mol/g}_{OC}$  should pose a low risk of adverse biological effects due to SEM metals. For sediments with high carbon-normalized  $SEM_x > 3,000 \mu\text{mol/g}_{OC}$ , adverse biological effects due to SEMs may be expected. For sediments with carbon-normalized  $SEM_x > 130 \mu\text{mol/g}_{OC}$ , there is uncertainty about whether effects are expected and additional study (e.g., toxicity tests) is recommended (USEPA 2005).

- **Other ions.** Cationic metals compete with calcium for binding sites, and also will readily bind with the oxides of magnesium and iron, thus altering their

bioavailability. Measurements of the concentrations of these ions in bulk samples will also be helpful in interpreting the potential for toxicity of these metals.

The application of AVS, SEM, and foc are practical steps in evaluating metal bioavailability based on bulk sediment characteristics that are relatively easy and routine to obtain. Additional information regarding relative COPC bioavailability will be gained by concomitant porewater characterization.

### **B1.2.1 Field Porewater Measurements**

Field porewater will be collected for analysis at the same time and location where sediments are collected. Field porewater data will help reduce uncertainty about potential risks associated with sediments and will be another line of evidence. Primary field porewater measurements to be conducted include the following:

- **Dissolved COPCs.** The dissolved concentration represents that fraction which passes through a 0.45 µm filter. For metals, the dissolved concentration provides a relevant measure of exposure because 1) national ambient water quality criteria (NAWQC) for metals are based on the dissolved concentration; 2) interstitial water toxicity unit (IWTU) methods are calculated by normalization of porewater concentrations by the NAWQC (USEPA 2005); and 3) dissolved concentrations may be interpreted in the context of the BLM as a way to account for the effects of water quality on metal bioavailability.
- **General chemical properties.** Standard measures necessary to help interpret COPC bioavailability include DOC, pH, and major cations/anions.

A BLM calculation based on sediment porewater composition (pwBLM) can explicitly account for the effects of DOC, pH, and cation concentrations on the bioavailability of porewater metals. The mechanistic basis of the pwBLM allows for an explicit consideration of mixtures based upon metals accumulated at biotic ligands as the basis for predicting biological effects.

### **B1.3 Whole-sediment Bioassays**

An additional measure of COPC bioavailability will include sediment bioassays synoptically performed on 53 percent of the samples (48 site samples and 26 internal, tributary, or upstream reference samples). These tests will provide direct measures that refine and reduce uncertainties regarding COPC toxicity and bioavailability. In addition, these tests will ascertain if Site sediments adversely affect the survival, growth, or reproduction of benthos. If significant differences are identified, these data will also

help to address 1) the magnitude of these effects; and 2) a concentration-response relationship between COPCs and observed effects. The collection of toxicity data will be coordinated with the collection of laboratory porewater chemistry data from test chambers to support test interpretation.

#### **B1.3.1 Biological Endpoints (Measurements)**

Seventy-four sediment samples will be used to conduct acute and chronic bioassays with *H. azteca* and *C. dilutus*. Four standard sediment toxicity tests will be conducted. Specifically, the following two bioassays will be performed on all 74 samples:

- 28-day whole-sediment toxicity tests with the amphipod, *H. azteca*
- 10-day whole-sediment toxicity tests with the midge, *C. dilutus*.

As noted within Section A7.3.2, to meet Study DQOs and minimize the potential for confounding inter-batch variability with other variables (e.g., due to a chemical gradient), short-term bioassay testing will be initiated only after completing all field sampling. Short-term bioassays will be conducted in multiple batches, with each batch consisting of up to approximately 15 samples plus controls. Samples will be assigned to batches using a stratified random approach. The strata will be based on river reaches to ensure that each batch will contain samples from across all geographic areas of the river (including external reference locations), to the maximum extent possible. Within strata, samples will be randomly selected for each batch. Upon identifying and assigning bioassay samples in respective batches, the stratified random bioassay batching scheme will be reviewed and approved by EPA prior to bioassay testing.

In addition, reproductive endpoints will be evaluated on 18 split-samples. Preference for these 18 split-samples will be given to sampling stations located within high-medium exposure gradients. Sample selection will be evaluated using results of the above-listed 10- and 28-day survival and growth tests in conjunction with preliminary chemistry data; and presented in a technical memorandum for EPA's review and concurrence. It is anticipated that sample selection will target sediment with 1) low to moderate toxicity response in short-term studies; 2) high metal concentrations in porewater or bulk sediment; and/or 3) a range of sediment and porewater characteristics. Specific bioassays to be performed on these 18 split-samples include the following:

- 42-day whole-sediment toxicity tests with the amphipod, *H. azteca*.
- 50- to 65-day whole-sediment toxicity tests with the midge, *C. dilutus*.

Standard responses (endpoints) of test organisms to be measured are summarized in Table B1-2 and include the following:

- Survival (number of surviving organisms divided by the initial number of organisms).
- Weight
  - *H. azteca*—dry weight [DW] of surviving organisms divided by the number of surviving organisms
  - *C. dilutus*—ash-free dry weight [AFDW] of surviving organisms divided by the number of surviving organisms.
- Biomass
  - *H. azteca*—DW of surviving organisms divided by the initial number of organisms
  - *C. dilutus*—AFDW of surviving organisms divided by the initial number of organisms
- Reproduction (measures of reproduction vary by bioassay and may include number of young divided by the number of females surviving bioassay; number of eggs oviposited divided by the number of females surviving; number of eggs produced divided by the number of females surviving, etc.).
- Emergence (applicable only to the long-term *C. dilutus* bioassay tests, measures of emergence include number of organisms that reach the terrestrial adult [imago] stage divided by the initial number of organisms; and the time until emergence).

Standard bioassay test conditions for the above-referenced four tests are in Tables B1-3 through B1-6 (USEPA 2000). Required performance criteria are in Tables B1-7 through B1-10 (USEPA 2000). Standard bioassay endpoints will be reported in accordance with applicable guidance (USEPA 2000; ASTM 2012) including those endpoints specific to long-term *C. dilutus* bioassays noted on Table 15.4 of USEPA (2000).

Sediments with a low  $mPECQ_{\text{metals}}$  (e.g., <0.2), may be re-assigned *a posteriori* as “internal” reference sites, in consultation with EPA. Designated internal and external references sites will be integrated into a reference envelope approach that will define a range, or lower tolerance limit, of acceptable reference conditions against which toxic sediment can be compared (Hunt et al. 2001).

### **B1.3.2 Physico-chemical Data in Overlying Water**

A variety of physico-chemical properties will be measured in the test chamber water column (overlying water) to document water quality during bioassay tests as specified by USEPA (2000); see Tables B1-3 to B1-6.

The following water quality properties will be documented in each of the test chambers:

- Hardness (mg/L as calcium carbonate)
- Alkalinity (mg/L as calcium carbonate)
- Conductivity ( $\mu\text{S}/\text{cm}$ )
- pH (standard units)
- Ammonia (mg/L)
- Temperature ( $^{\circ}\text{C}$ )
- Dissolved oxygen (mg/L).

### **B1.3.3 Laboratory Porewater Measurements**

Laboratory porewater will be collected at the beginning and end of the bioassay tests for the short-term tests and at the beginning, midpoint, and end of the tests for the long-term tests. Laboratory porewater data will be used in concert with the biological endpoint data to evaluate concentration-response relationships. Primary laboratory porewater measurements (volume permitting) will include the following:

- **Dissolved COPCs.** For metals, the dissolved concentration provides a relevant measure of exposure because 1) NAWQC for metals are based on the dissolved concentration; 2) IWTU methods are calculated by normalization of porewater concentrations by the NAWQC (USEPA 2005); and 3) dissolved concentrations may be interpreted in the context of the BLM as a way to account for the effects of water quality on metal bioavailability.
- **General chemical properties.** Standard measures necessary to help interpret COPC bioavailability include DOC, pH, and major cations/anions.

A pwBLM can explicitly account for the effects of DOC, pH, and cation concentrations on the bioavailability of porewater metals. The mechanistic basis of the pwBLM allows for an explicit consideration of mixtures based upon metals accumulated at biotic ligands as the basis for predicting biological effects.

### **B1.4 Toxicity Identification Evaluation**

Should equivocal or unexplained differences be identified during the whole-sediment bioassays, further evaluation using TIE could be completed on select samples to address if the effects are due to classes of COPCs. Samples selected for TIEs would be identified in a technical memorandum; see Section B4.2.2 for decision rules regarding sample selection. TIEs would be performed in accordance with EPA guidance and studies reported in the scientific literature (e.g., Ho et al. 2007; Hockett and Mount 1996).

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## **B2      SAMPLING METHODS**

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Field sampling methods for collection of bulk chemistry and porewater samples are described in the FSP (Appendix A). The FSP includes the following topics:

- Station positioning (Section 2.2.2)
- Field equipment and supplies (Section 2.2.3)
- Sampling methods (Section 2.2.3.1)
- Sample containers and labels (sample labels, sample identifier custody seals, sample custody/tracking procedures) (Section 2.5)
- Field documentation and procedures (field logbooks, photo documentation, COC forms) (Section 3).

SOPs for each sampling method are provided in Attachment 2 of the FSP.

The FSP also describes the collection of field split samples that will be provided to EPA for analysis as part of their QA/QC program. These will contain not less than 200 grams of sediment and will comprise approximately 15 percent of the samples collected for chemical analysis. In addition, up to seven split samples from bioassay stations located upstream from the confluence of Onion Creek (RM 730) will also be evaluated as part of EPA's QA/QC program. Pending approval and agreement from the Canadian Government, EPA would also collect up to three split-samples for bioassay testing in upstream reference locations. Field QC samples are described in Section 2.2.3.4 of the FSP.

In the event that unanticipated or changed circumstances occur in the field, the field supervisor will institute the necessary corrective actions, complete a corrective action record, and ensure that the appropriate procedures are followed. If corrective actions require a departure from the FSP, these changes will be documented on a field change request form (refer to Appendix A for examples of these and other forms) and submitted to EPA. In any other circumstances where sampling conditions are unexpected, the appropriate sampling actions consistent with this task's objectives will be conducted. This change will be noted by the field supervisor in the field log, and a change request form will be completed for the project files and submitted to EPA. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the TAI technical team coordinator, TAI project coordinator, and EPA. EPA will be notified of any problems that may affect the final outcome of this task. Additional information regarding corrective actions and related documentation is provided in Section C1.

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### **B3 SAMPLE HANDLING AND CUSTODY**

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Principal documents used to identify samples and to document possession will be field logbooks and COC records. Custody will be documented for all samples at all stages of the analytical or transfer process. COC procedures for sample handling prior to delivery to the laboratories are outlined in the FSP (Appendix A).

Upon receipt of samples, the laboratory will check the physical integrity of the containers and custody seals, and samples will be inventoried by comparing sample labels to those on the COC forms. The laboratory will include the COC and shipping container receipt forms in the data package. Any breaks in the COC or non-conformances will be noted and reported in writing to the laboratory coordinator within 24 hours of receipt of the samples. Specific laboratory QA plans are provided in Appendix D (analytical laboratory) and Appendix E (bioassay laboratory). Laboratory project managers will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratory.

Sediment samples will be stored in accordance with specifications detailed in Table B3-1; storage specifications for porewater samples are in Table B3-2. Laboratories will maintain COC documentation and documentation of proper storage conditions for the entire time that the samples are in their possession. The laboratory will not dispose of the samples for this task until authorized to do so by the task QA coordinator.

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### **B4 SAMPLE PROCESSING AND ANALYTICAL METHODS**

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Samples collected for this study will be analyzed for chemical parameters shown in Table A7-2 as summarized below.

#### **B4.1 Chemical Analyses**

COPC concentrations in whole-surface sediments will be measured and samples will be characterized for grain size, organic carbon content, AVS, SEM, TAL metals, and other parameters as appropriate (e.g., pH). Field porewater will be collected using airstones (refer to Section A7.3.1 and Appendix A for method) and preserved for the following analyses (volume permitting): dissolved TAL metals, pH, DOC, hardness, alkalinity, and major cations/ions (calcium, magnesium, sodium, chloride, potassium, and sulfate). Table B3-2 includes order of priority for these analyses. AVS and SEM will be measured in at least one chemistry-only replicate per sample during sediment toxicity tests (including repeat measurements during long-term reproduction toxicity tests). Bulk sediment chemistry, porewater metals (from peepers), and BLM parameters (from

centrifuged sediment) will be analyzed anew prior to longer-term reproduction toxicity tests.

## B4.2 Bioassays

Bioassay methodologies and protocols to be employed will be similar for the test species (*H. azteca* and *C. dilutus*) following the standard protocols outlined below. Details are described in EPA (USEPA 2000) and ASTM (2012).

Bioassay endpoints will be evaluated using a minimum of 8 replicates for biological endpoints per sediment sample for each short-term bioassay (Figure B4-1), and a minimum of 12 (42-day *H. azteca*) or 16 (long-term *C. dilutus*) replicates for biological endpoints for each long-term bioassay (Figure B4-2). Additional replicate bioassay chambers will be run on each sediment sample exclusively to assess porewater. Chemistry replicates will not be used to evaluate biological endpoints (i.e., survival, growth, or reproduction). Thus, the 28-day *H. azteca* bioassays will have a total of 14 replicates (8 for biological endpoints and 3 each for porewater chemistry analysis at day 7 and during the week prior to day 28). The 10-day *C. dilutus* assays will have 11 replicates for the 10-day test (8 for biological endpoints and 3 for porewater chemistry analysis at day 7). The 42-d *H. azteca* bioassay will have a total of 18 replicates (12 for biological endpoints and 3 each for porewater chemistry analysis at day 7 and sometime between days 21 and 27). The long-term *C. dilutus* bioassays will have a total of 25 replicates (16 for biological endpoints and 3 each for chemistry analysis at day 7, sometime between days 21 and 27, and again between days 42 and 49). The 16 biological replicates specified for the long-term *C. dilutus* bioassay includes four test chambers that will be run solely to produce auxiliary males for possible use in the bioassay test. These chambers are not true test replicates and will not be assessed for biological endpoints. Schematics illustrating the above-mentioned anticipated number of bioassay and chemistry-only replicates are presented in Figures B4-1 and B4-2, and the total number of replicate chambers is shown in tabulated form in Table B4-1.

Prior to bioassay testing, sediment samples will be homogenized, and 100 mL of the sediment will be distributed into each replicate and covered with laboratory water. Test chambers will be allowed to stabilize for one day prior to the introduction of test organisms. From the laboratory culture population, 10 test organisms (except for long-term *C. dilutus* tests which have 12 test organisms) will be randomly distributed to each replicate and allowed to burrow into the sediment.

Water quality will be measured in the overlying water of representative replicate chambers for each sample according to EPA guidance. Lighting, room temperature, and



other environmental operations of the exposure system will be monitored daily. As required in USEPA (2000) and ASTM (2012) (and listed in Tables B1-3 to B1-10), conductivity, hardness, pH, alkalinity, and ammonia will be measured in the overlying water of test chambers at the initiation and termination of the bioassays. Conductivity will also be measured weekly, and DO and ammonia on a daily basis. Dissolved oxygen will be maintained above 2.5 mg/L; water temperature will be measured daily in at least one test replicate per treatment to ensure that the daily average temperature is within  $\pm 1^\circ\text{C}$  of  $23^\circ\text{C}$ .

At test termination, survival, weight, biomass, and any other required endpoints will be assessed and recorded. Endpoints for each bioassay are listed in Table B1-2.

#### **B4.2.1 Laboratory Porewater Analysis**

The additional chambers set-up for chemistry analysis of each sediment sample will contain test organisms, but will only be used for porewater chemistry measurements. Porewater will be sampled from each sediment sample selected for short-term toxicity tests at the start of exposures using centrifugation. These porewater samples will be analyzed for DOC, pH, alkalinity, sulfide, major cations, and major anions to inform the BLM for interpreting toxicity data. Porewater will also be collected using Brumbaugh type peepers; refer to SOP-9 of Appendix A. Porewater collected from the Brumbaugh type peeper will be analyzed for TAL metals except for mercury.

#### **B4.2.2 Toxicity Identification and Evaluation (TIE)**

Should equivocal or unexplained differences be identified during the whole-sediment bioassays or if the calculated concentration-response curve is not robust, further evaluation using TIE will be completed on selected samples to address if the effects are due to a class of COPCs. It is not possible to determine *a priori* which samples might need to undergo TIE testing. In addition to the equivocal samples, it will be desirable to analyze a few toxic and non-toxic samples where the toxicity results correlate well with contaminant concentrations to ensure the TIE tests are performing as expected. A technical memorandum will be prepared detailing which samples will be tested, why those samples were selected, and the TIE test procedures to be used. The following factors will be considered in determining whether to run TIE tests:

- The robustness of the correlation supporting a stressor-response gradient. If an extremely robust gradient of correlated exposure and response is observed, the TIE will not be conducted as it may not be that important to identify the specific cause of toxicity. However, if the gradient is weak or if correlations are less definitive, then a TIE would help reduce uncertainty and will be performed.

- If there are significant outliers from the general correlations between exposure variables and effects. There may be more than one exposure variable across the UCR that can induce sediment toxicity, and these may or may not correlate with one another. In addition, it is always possible that some sediment characteristic could cause effects in sediment toxicity tests, but that characteristics would not be readily captured by the sediment characteristics that are being measured.
- Strength of the toxicity response in the bioassays. Less toxicity will mean it is less likely a TIE will be successful in identifying the toxicant, so samples with marginal toxicity will not be analyzed.

The following Phase I methodologies could be implemented:

- **Zeolite addition to evaluate ammonia toxicity.** Based on available guidance (Ho et al. 2007; Hockett and Mount 1996), a 20 percent (v/v or wwt/wwt) Zeolite sediment addition is adequate to evaluate the ammonia-caused toxicity. The appropriate amount of Zeolite would be mixed thoroughly into the sediment and allowed to equilibrate for 1 to 4 days prior to organism addition and bioassay initiation, as detailed in Ho et al. (2007).
- **Cation exchange resin or sulfide addition to reduce soluble metals.** Available guidance literature suggest the use of a SIR-300 resin, which exhibits a high affinity for copper, cadmium, zinc, nickel, and lead; once prepared, a 20 percent addition of resin to sediment is recommended, followed by a minimum 24-hour equilibrium period. Alternatively, sulfide addition is accomplished by spiking sediments with a sodium sulfide (hydrate form) solution (Ho et al. 2007).
- **Coconut charcoal/carbonaceous resin addition to reduce toxicity of organic chemicals.** Available guidance literature recommends that the rate of charcoal addition depends on the physical properties of sediment (2 percent to 5 percent for fine and medium sediments); alternatively, a 20 percent addition of Ambersorb (wwt/wwt) resin can also be used (Ho et al. 2007).

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## **B5 QUALITY CONTROL**

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Laboratory QC procedures are described below.

### **B5.1 Analytical Laboratory Quality Control**

Extensive and detailed requirements for laboratory QC procedures are provided in the EPA methods that will be used for this study (Table A7-2). Every method protocol includes descriptions of QC procedures, and many incorporate additional QC

requirements by reference to separate QC sections. QC requirements include control limits and requirements for corrective action in many cases. QC procedures will be completed by the laboratories, as required in each protocol and their internal SOPs, and as indicated in this QAPP.

The frequency of analysis for LCSs, MS/MSD samples or laboratory duplicates, and method blanks will be one for every 20 samples or one per extraction or analysis batch, whichever is more frequent. Calibration procedures will be completed at the frequency specified in each method description. Equipment blanks will be subjected to the same processes as the sediment preparation.

As required for EPA SW-846 methods (USEPA 2008), performance-based control limits have been established by the laboratory. These and all other control limits specified in the method descriptions will be used by the laboratory to establish the acceptability of the data or the need for reanalysis of the samples. Laboratory control limits for recovery of internal standards (including certified reference material), matrix spikes, and LCSs, and for relative percent difference of laboratory duplicates, are provided in the analytical laboratory's QA manual (Appendix D).

## B5.2 Data Quality Indicators

The overall quality objective for this task is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. QA procedures and measurements that will be used for this task are based on EPA guidance. Data quality indicators such as the PARCC parameters and analytical sensitivity will be used to assess conformance of data with QC criteria (USEPA 2002b). Measurement quality objectives (MQOs) for the quantitative PARCC parameters are provided in Tables B5-1 and B5-2. Data quality indicators and QC objectives are described in this section.

**Precision** reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of laboratory duplicates and field splits. Precision is expressed in terms of the RPD for two measurements. The following equation is used to calculate the RPD between measurements:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where: RPD = relative percent difference  
C<sub>1</sub> = first measurement  
C<sub>2</sub> = second measurement

For three or more measurements, the relative standard deviation (RSD) is used to evaluate precision. The RSD is calculated as the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

**Accuracy and bias** represent the degree to which a measured concentration conforms to a reference value. Results for matrix spikes, LCSs, field blanks, and method blanks will be reviewed to evaluate accuracy and bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = \frac{M - U}{C} \times 100$$

Where:            %R = percent recovery  
                      M = measured concentration in spiked sample  
                      U = measured concentration in unspiked sample  
                      C = concentration of added spike

Percent recovery for a LCS or reference material is calculated as follows:

$$\%R = \frac{M}{C} \times 100$$

Where:            %R = percent recovery  
                      M = measured concentration in reference sample  
                      C = established reference concentration

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Detection of any target analytes in field or method blanks will be evaluated as potential indicators of bias.

QC samples and procedures are specified in each method protocol (analytical methods are presented in Table A7-2). All QC requirements will be completed by the analytical laboratories as described in the protocols, including the following (as applicable to each analysis):

- Initial calibration
- Initial calibration verification
- Continuing calibration
- Calibration or instrument blanks
- Method blanks
- Laboratory control samples
- Internal standards (including certified reference material)

- Serial dilutions
- Matrix spikes
- Laboratory duplicates.

To alert data users of possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated QC samples or procedures do not meet laboratory internal control limits (Appendix D).

Analytical concentration goals (ACGs) provide the target concentration required for the chemical analysis. Methods selected for this study are expected to provide sufficient sensitivity to yield ACGs that are below the lowest reference value for this study (Table A7-3).

The laboratory will determine a MDL for each analyte, as required by EPA (USEPA 2004). MDLs are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix with 99 percent confidence that a false positive result has not been reported. The analytical laboratory will have established MRLs at levels above the MDLs for the task analytes. These values are based on the laboratory's experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system; they represent the level of analyte above which concentrations are accurately quantified.

The laboratory will quantify analytes at concentrations above the MRL. Analytes detected at concentrations between the MDL and MRL will be flagged with a "J" qualifier to indicate that the value is an estimate (i.e., the analyte concentration is greater than or equal to the MDL and less than the MRL). Analytes that are not detected will be reported as the MDL and will be flagged with a "U" qualifier. MDLs will be adjusted by the laboratory as necessary to reflect sample dilution or matrix interference.

**Representativeness** is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratory, representativeness will be ensured by the proper handling and storage of samples, the use of standard performance-based methods, and initiation of analyses within holding times.

**Comparability** is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by EPA.

**Completeness** is a measure of the amount of valid data obtained from the analytical measurement system and the complete implementation of defined field procedures. The target completeness objective will be 90 percent; the actual completeness may vary depending on the intrinsic nature of the samples. Completeness of the data will be assessed during QC reviews.

Completeness is defined as follows for all measurements:

$$\%C = \frac{V}{T} \times 100$$

Where:        %C = percent completeness  
                 V = number of measurements judged valid  
                 T = total number of measurements

## **B6     INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND          MAINTENANCE**

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Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the laboratories in accordance with requirements identified in laboratory SOPs and manufacturer instructions. In addition, each of the specified analytical methods provides protocols for proper instrument setup, tuning, and critical operating parameters. Instrument maintenance and repair will be documented in the laboratory's maintenance logs or record books.

## **B7     INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY**

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Before beginning each analysis, laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratories' QA plans.

Calibration standards will be obtained from either the EPA repository or a commercial vendor, and the laboratories will maintain traceability back to the National Institute of Standards and Technology (NIST). Stock standards will be used to establish intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be

checked against standards from another source, as specified in the methods and the laboratory QA manual.

## **B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES**

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The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and quality control purposes.

The quality of laboratory water used will be documented at the laboratory. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in laboratory SOPs and QA plans. All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by the field supervisor (i.e., for supplies used in the field) or the laboratory QA manager (i.e., for supplies used in the laboratory).

## **B9 DATA MANAGEMENT**

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Data for this study will be generated both in the field and at the analytical and bioassay laboratories. The final repository for sample information will be the relational database housed at <http://teck-ucr.exponent.com>. Procedures used to transfer data from the point of generation to the database are described in this section.

The data management plan (DMP) and its amendment establishes standard procedures for the management of all documents and environmental data (field and laboratory) generated during the RI/FS. The DMP describes data management procedures relating to the creation, acquisition, handling, storage, and distribution of task-related data. Data management systems and procedures described below are intended to establish and maintain an efficient organization of large volumes of complex environmental information for a diverse combination of data types. To accomplish this task, the following four management systems will be used to provide organized and efficient data management and retrieval:

- **Project database.** Stores environmental sampling and analysis data, information pertaining to geographic information system (GIS) files, and citations of documents related to collection, analysis, or interpretation of environmental data stored in the database. Both current and historical data are stored in the project database.
- **Geographic information system (GIS).** Stores spatial data and enables the cartographic presentation of data trends and patterns.
- **Hard copy files.** Maintains a record and archive of documents from field studies and resulting reports.
- **Web site** (<http://www.ucr-rifs.com>). Makes available draft documents and other project information via the secure domain. Users with appropriate privileges are able to download documents.

Study activities will use spatial data sets and analyses for planning, data interpretation, decision support, and data presentation. Links between data in the project database and GIS files will be established via common identifiers for sampling locations and other geographic features.

### **B9.1 Field Data**

Data that are generated during sediment collection and sample preparation will be manually entered into the field logbook, field data forms, and COC forms. Data from these sources will be entered into the project database directly from the field logbook and field data forms. These data include sample collection coordinates, station names, sampling dates, sample identifiers and numbers, and additional station and sample information. All entries will be reviewed for accuracy and completeness by a second individual, and any errors will be corrected before the data are approved for release to data users.

### **B9.2 Analytical Laboratory Data**

A variety of manually entered and electronic instrument data will be generated at the laboratories. Data will be manually entered into the following:

- Standard logbooks
- Storage temperature logs
- Balance calibration logs
- Instrument logs



- Sample preparation and analysis worksheets
- Maintenance logs
- Individual laboratory notebooks.

All manual data entry into the laboratory information management system will be proofed at the analytical laboratories. Data collected from each laboratory instrument, either manually or electronically, will be reviewed and confirmed by analysts before reporting. A detailed description of procedures for laboratory data management and data review and verification is provided in the laboratory QA plans (Appendices D and E).



## **SECTION C: ASSESSMENT AND OVERSIGHT**

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This task will rely on the knowledge and expertise of the TAI technical team. The field team and laboratories will stay in close verbal contact with the co-principal investigator and the task QA coordinator during all phases of this task. This level of communication will serve to keep the management team apprised of activities and events, and will allow for informal but continuous task oversight.

### **C1 ASSESSMENTS AND RESPONSE ACTIONS**

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Assessment activities will include readiness reviews prior to sampling and prior to release of the final data to the data users, as well as internal review while work is in progress. An informal technical systems audit may be conducted if problems are encountered during any phase of this task.

Readiness reviews are typically conducted to ensure that all necessary preparations have been made for efficient and effective completion of each critical phase of work. The first readiness review will be conducted prior to field sampling. The field supervisor will verify that all field equipment is ready for transfer to the site. The field supervisor will also verify that the field team and subcontractor(s), as required, have been scheduled and briefed, and that the contract for the subcontractor has been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed before final data are released for use. The database administrator will verify that all results have been received from the laboratories, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the database administrator, the task QA coordinator, or their designee. Data will not be released for final use until all data have been verified and validated and approved by EPA. No report will be prepared in conjunction with the readiness reviews.

Technical review of intermediate and final work products generated for this task will be completed throughout the course of all sampling and laboratory activities, data validation, data management, and data interpretation to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the TAI technical

team coordinator and TAI project coordinator. EPA will be notified of any problems that may affect the final outcome of this task, according to the Agreement.

The laboratories will be required to have implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Each phase of work will be reviewed by a supervisor before it is approved for release. Details are provided in the laboratory QA plans (Appendices D and E).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. Any task team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance to the co-principal investigator, the task QA coordinator, or the laboratory project or QA manager, as applicable. The task QA coordinator will ensure that no additional work dependent on the non-conforming activity is performed until a confirmed non-conformance is corrected. Any confirmed non-conformance issues will be relayed to the TAI technical team coordinator. In addition, during corrective actions, communication between the field personnel and the laboratory relative to the accuracy and completeness of the COC documents will follow corrective-action procedures.

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## **C2      REPORTS TO MANAGEMENT**

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The laboratories will keep the appropriate technical team laboratory coordinator(s) and QA manager(s) apprised of their progress on a regular basis. The laboratories will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of out-of-control laboratory QC data that resulted in a requirement for corrective action and a description of the corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

The technical team laboratory coordinator and QA manager will provide this information to the task QA coordinator who, in turn, will provide this information to the TAI technical team coordinator.

The laboratory will be required to have implemented routine systems of reporting non-conformance issues and their resolution. These procedures are described in the laboratory QA manuals. Laboratory non-conformance issues will also be described in the field sampling report if they affect the quality of the data.

Data packages and EDDs will be prepared by the laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances (if applicable), and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Validated data will be provided electronically to EPA. These data will also be provided with the data summary report containing an overview of the field event, a sampling location map, sample collection methods, and rationale for any deviations from the FSPs and QAPP according to the Agreement.



## **SECTION D: DATA VALIDATION AND USABILITY**

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Data generated in the field and at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the data validation report. In the following sections, the term “laboratory” refers to both the analytical and bioassay laboratories.

### **D1 DATA REVIEW, VERIFICATION, AND VALIDATION**

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Field and laboratory data for this task will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected and documented prior to release of the final data.

Data verification and validation will be completed according to methods described in the following EPA guidance documents for data validation:

- Guidance on Environmental Data Verification and Validation (USEPA 2002b)
- EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (USEPA 2004).

Data will be qualified as estimated as necessary if results for surrogates, LCSs, MS/MSD samples, or laboratory duplicates do not meet method-specified control limits, including performance-based control limits. Results for other QC procedures will be qualified if they do not meet control limits outlined in EPA’s functional guidelines and SOPs for data validation (USEPA 1995, 1996, 1999, 2004). Data will be qualified as undetected based on concentrations of target analytes detected in laboratory or field blanks, according to EPA’s functional guidelines and SOPs for data validation.

Performance-based control limits are established periodically by the laboratories as required for the selected methods. Current values will be provided in the laboratory QA plans, as applicable.

No guidelines are available for validation of data for AVS, TOC, and DOC. These data will be validated using procedures described in the functional guidelines for inorganic data review (USEPA 2004), as applicable. Data will be qualified as estimated, as necessary, if results for QC samples do not meet performance-based control limits.

Results for field split-samples will be evaluated using control limits of 35 percent. Data will not be qualified as estimated if the MQOs are exceeded, but RPD results will be tabulated and any exceedances will be discussed in the data summary report.

Equipment rinse blanks will be evaluated and data qualifiers applied in the same manner as method blanks, described in the functional guidelines for data review (USEPA 1995, 1996, 1999, 2004). Data will be rejected if control limits for acceptance of data are not met, as described in USEPA (1995, 1996, 1999, 2004).

## **D2 VERIFICATION AND VALIDATION METHODS**

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Field data will be verified during preparation of samples and COC forms. Field data and COC forms will be reviewed daily by the field supervisor. After field data are entered into the project database, 100 percent verification of the entries will be completed to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is released for use.

Approximately 10 percent of the chemistry data will be fully validated, including the first two data packages generated for each chemical analysis type. Validation for the remaining data will be based on review of the summary forms for sample and QC data. If problems or questions are encountered during validation, the laboratory will be contacted for resolution. An additional full validation will be completed, if required, to fully assess the quality of the data or to verify that laboratory errors have been addressed.

Procedures for verification and validation of laboratory data and field QC samples will be completed as described in the functional guidelines and SOPs for data validation (USEPA 1995, 1996, 1999, 2004) and summarized in Section D1 above. Accuracy and completeness of each data set will be verified at the laboratory when EDDs are prepared and again as part of data validation. Ten percent of entries to the database from the laboratory EDDs will be checked against the hard-copy data packages. Data validation will be completed by ESI.

In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use.

MRL goals for this task are provided in Table A7-3. Reporting limits for non-detects will be compared to the MRL goals to evaluate method sensitivity for each sample. Any exceedance of actual MRLs over the target MRLs will be discussed in the data validation report.



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### **D3 RECONCILIATION WITH USER REQUIREMENTS**

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The goal of data validation is to determine the quality of each data result and to identify those that do not meet the task MQOs. Non-conforming data may be qualified as estimated (i.e., a “J” qualifier will be applied to the result) or rejected as unusable (i.e., an “R” qualifier will be applied to the result) during data validation if criteria for data quality are not met. Data may also be qualified as undetected during validation based on laboratory and field blank results. Rejected data will not be used for any purpose. A summary of the qualified data and the reasons for qualification will be included in the data validation report.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. However, these data are less precise or less accurate than unqualified data. Data users are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses. The data quality discussion in the data validation report will include information regarding the direction or magnitude of bias or the degree of imprecision for qualified data to facilitate the assessment of data usability. Data validation reports will also include a discussion of data limitations and their effect on data interpretation activities.



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## FIGURES

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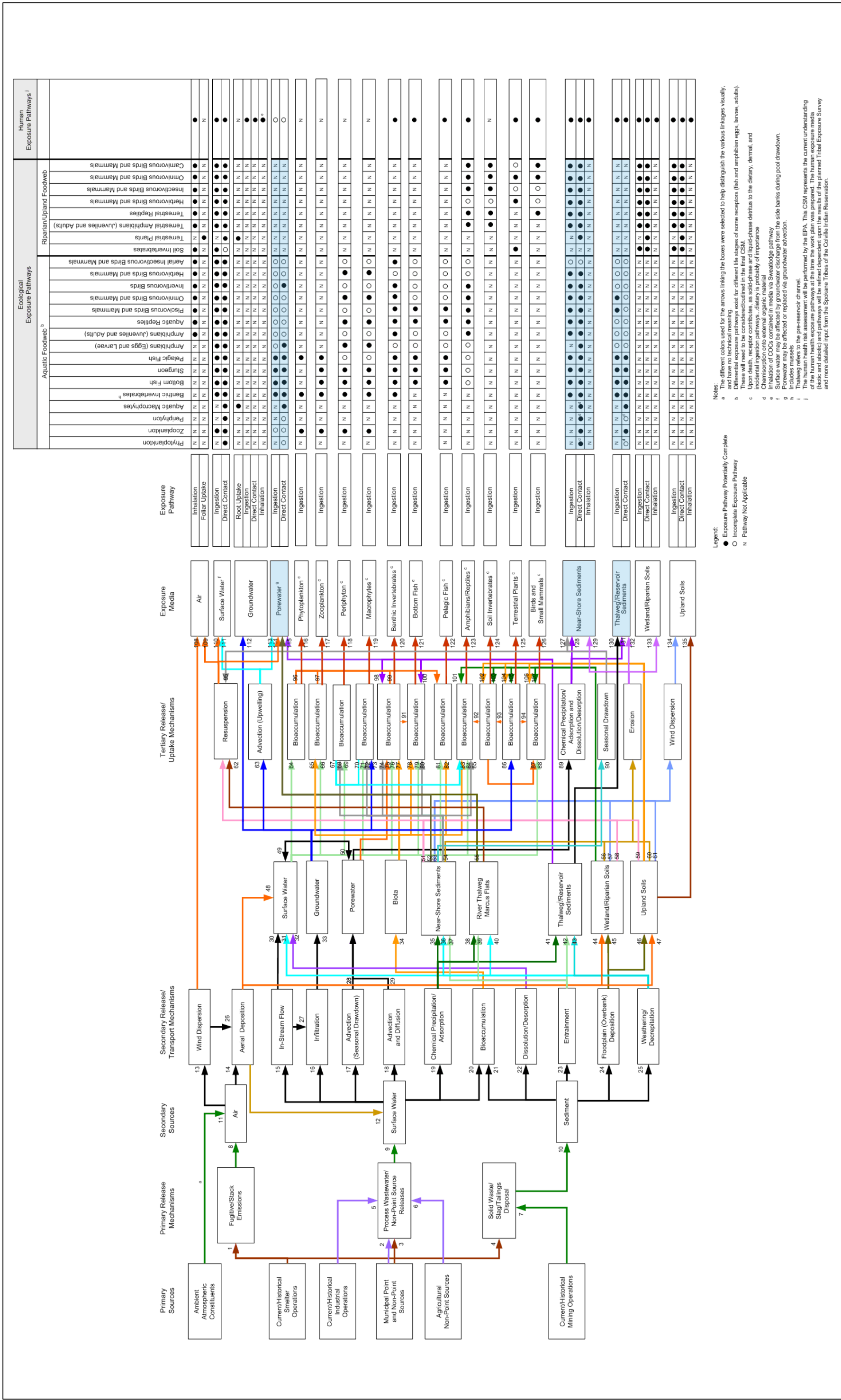
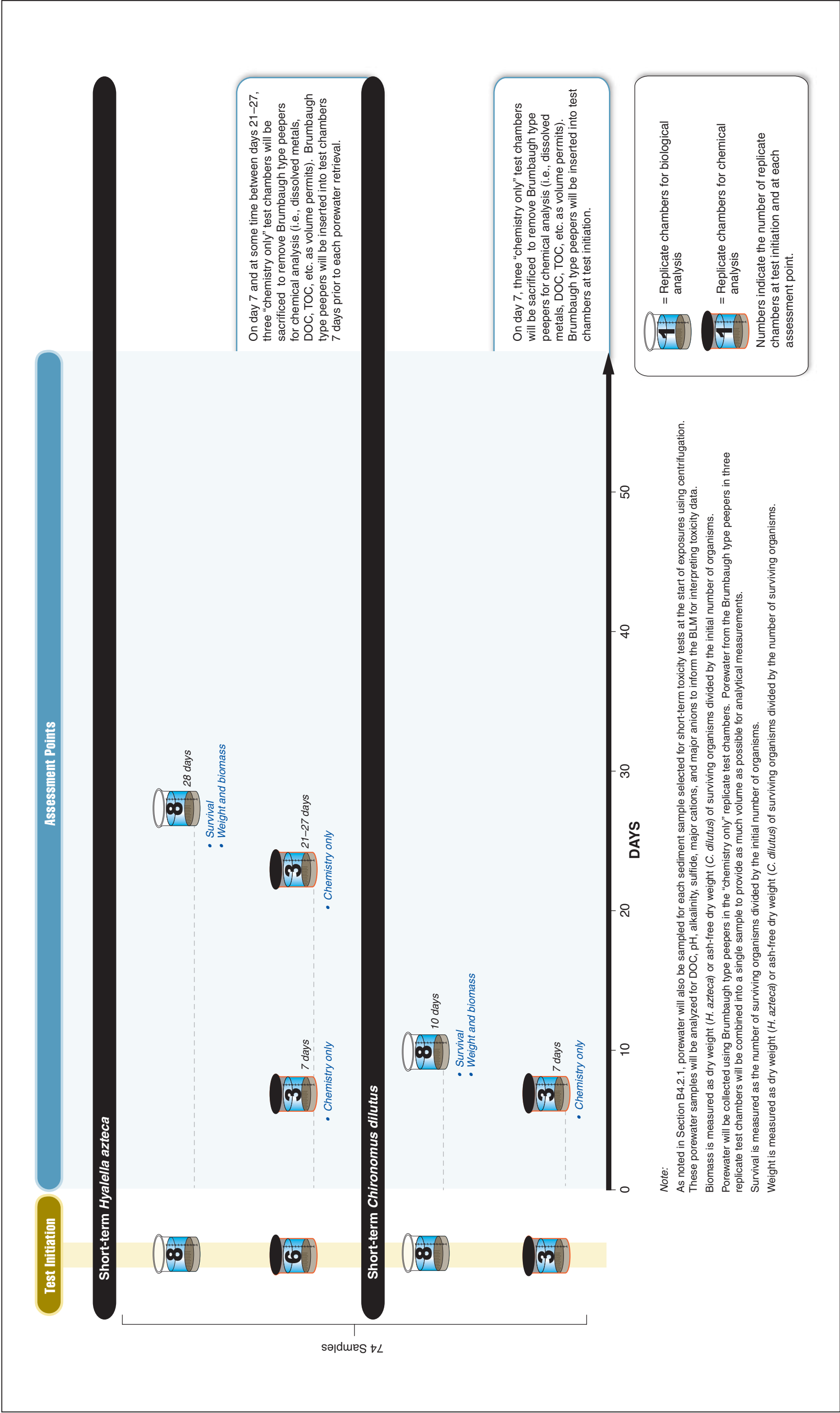


Figure A6-1. Sitewide Conceptual Site Model

Note: Exposure media and ecological receptors that Phase 2 Sediment Study data will address are shaded in blue.

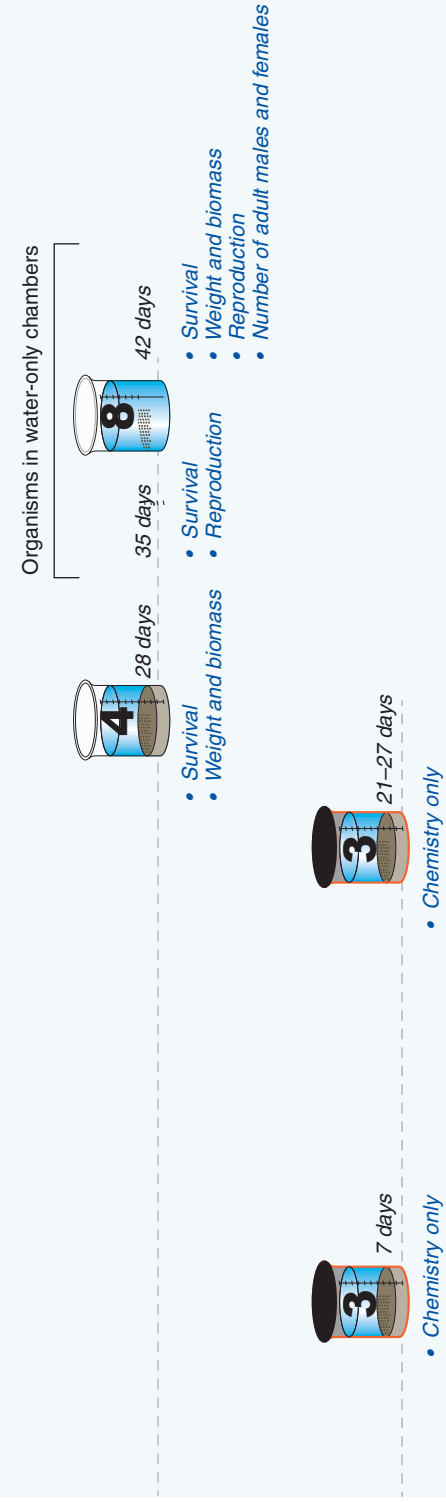




Test Initiation

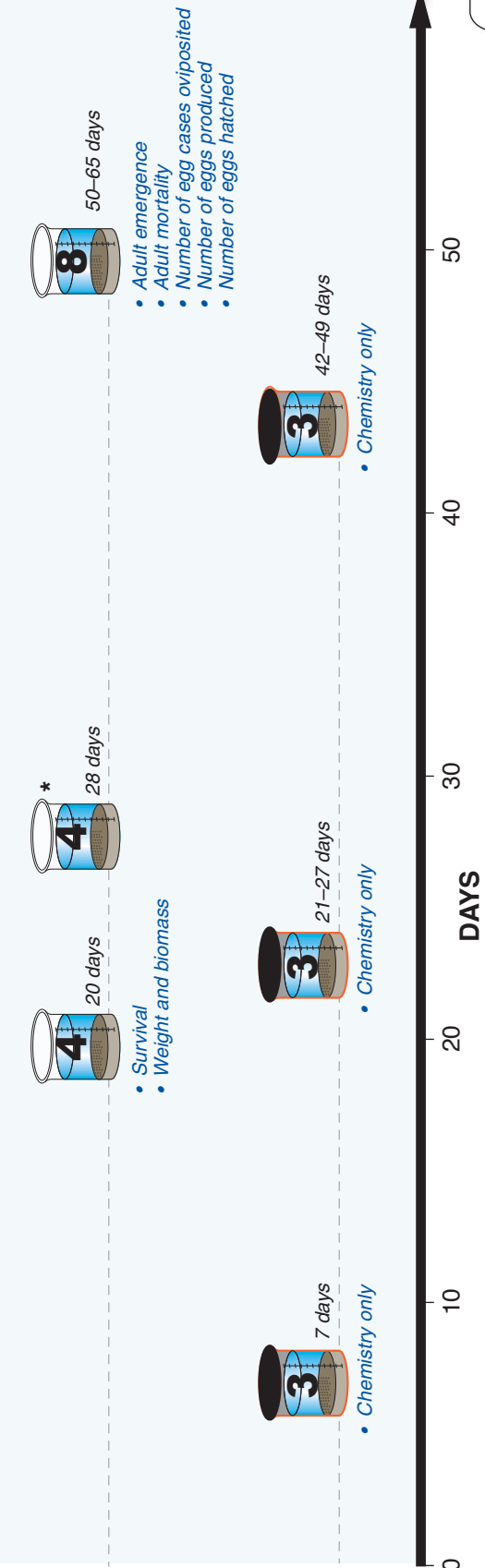
Assessment Points

Long-term *Hyalella azteca*



On day 7 and at some time between days 21–27, three “chemistry only” test chambers will be sacrificed to remove Brumbaugh type peepers for chemical analysis (i.e., dissolved metals, DOC, TOC, etc. as volume permits). Brumbaugh type peepers will be inserted into test chambers 7 days prior to each porewater retrieval.


Long-term *Chironomus dilutus*



On day 7 and at some time between days 21–27 and days 42–49, three “chemistry only” test chambers will be sacrificed to remove Brumbaugh type peepers for chemical analysis (i.e., dissolved metals, DOC, TOC, etc. as volume permits). Brumbaugh type peepers will be inserted into test chambers 7 days prior to each porewater retrieval.



= Replicate chambers for biological analysis



= Replicate chambers for chemical analysis

Numbers indicate the number of replicate chambers at test initiation and at each assessment point.

Note:

As noted in Section B4.1, bulk sediment chemistry, porewater metals (from peepers), and BLM parameters (from centrifuged sediment) will be analyzed anew prior to longer-term reproduction tests.

Biomass is measured as dry weight (*H. azteca*) or ash-free dry weight (*C. dilutus*) of surviving organisms divided by the initial number of organisms.

Porewater will be collected using Brumbaugh type peepers in the “chemistry only” replicate test chambers. Porewater from the Brumbaugh type peepers in three replicate test chambers will be combined into a single sample to provide as much volume as possible for analytical measurements.

Survival is measured as the number of surviving organisms divided by the initial number of organisms.

Weight is measured as dry weight (*H. azteca*) or ash-free dry weight (*C. dilutus*) of surviving organisms divided by the number of surviving organisms.

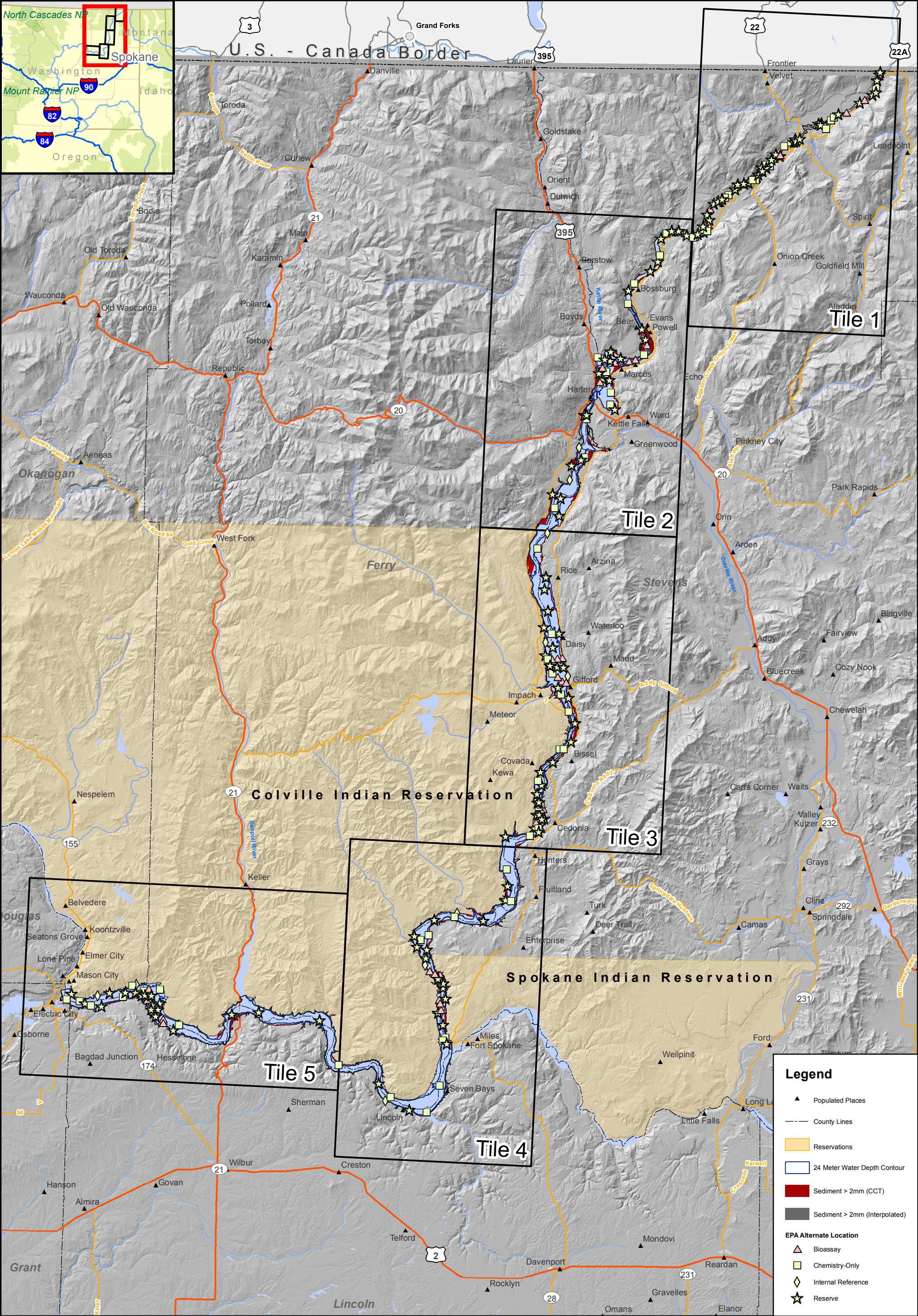
\* Four test chambers will be populated with *C. dilutus* in order to produce auxiliary males beginning on Day 28. These males may be used in the latter part of the long-term *C. dilutus* bioassay test. These test chambers are not true replicates and will not be measured for biological endpoints.

Figure B4-2. Long-term Bioassay Timeline for Each Sediment Sample

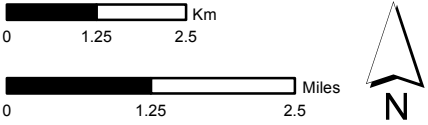
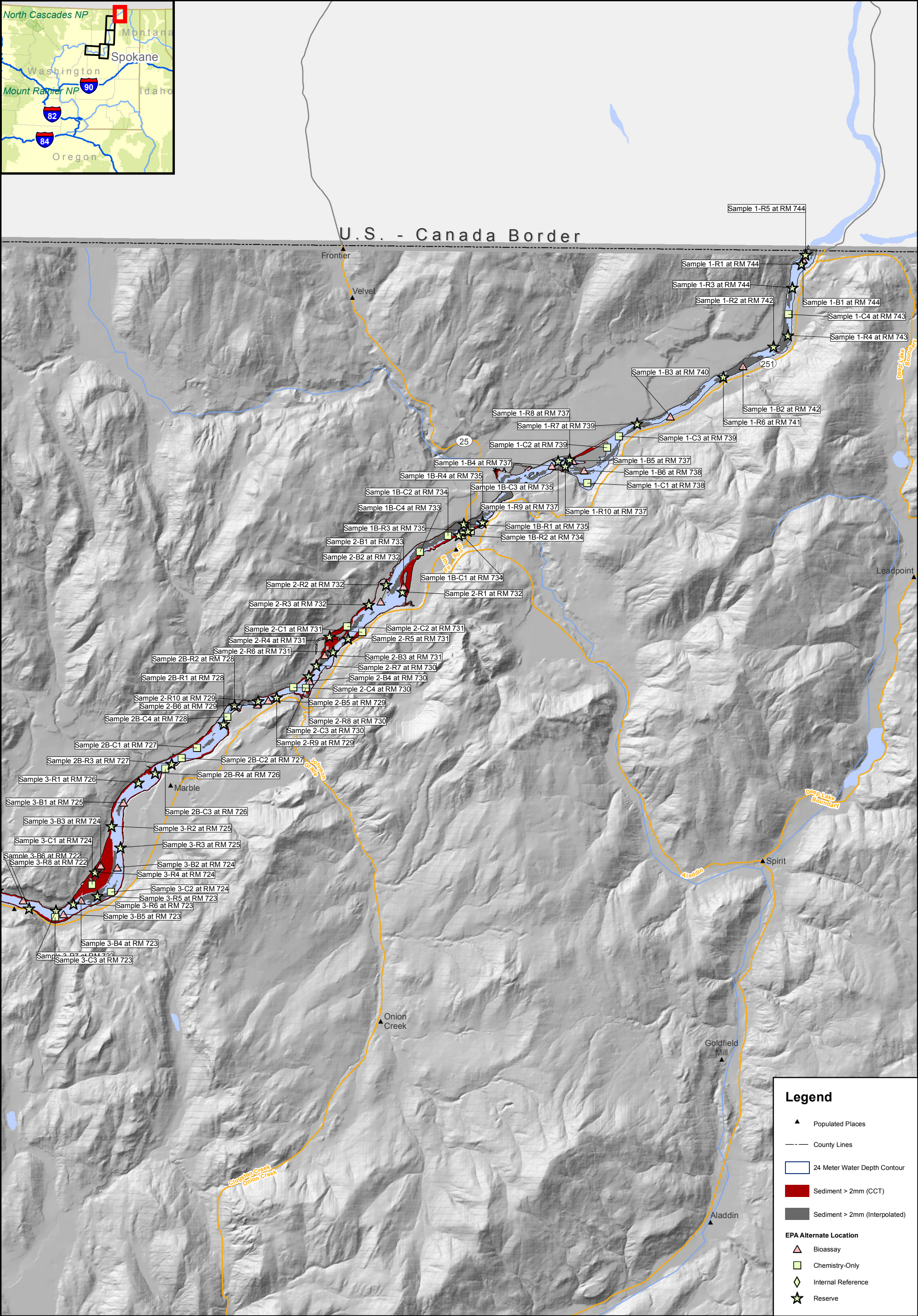
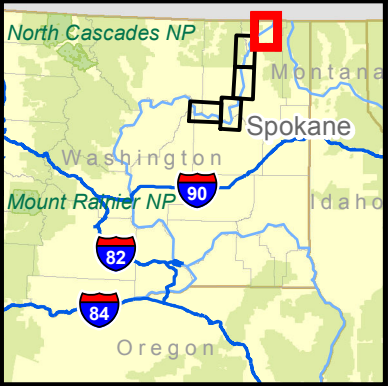
## MAPS

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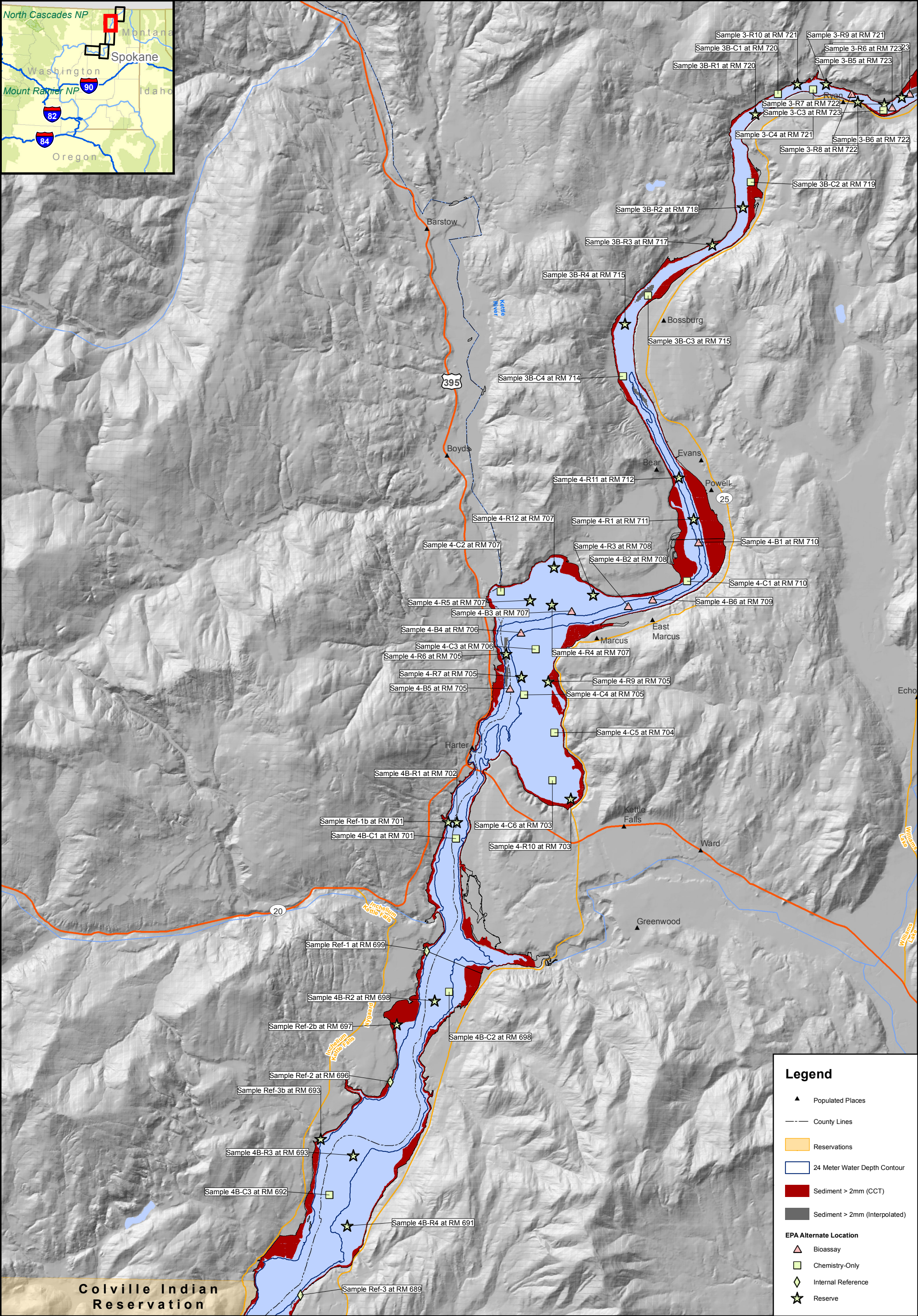




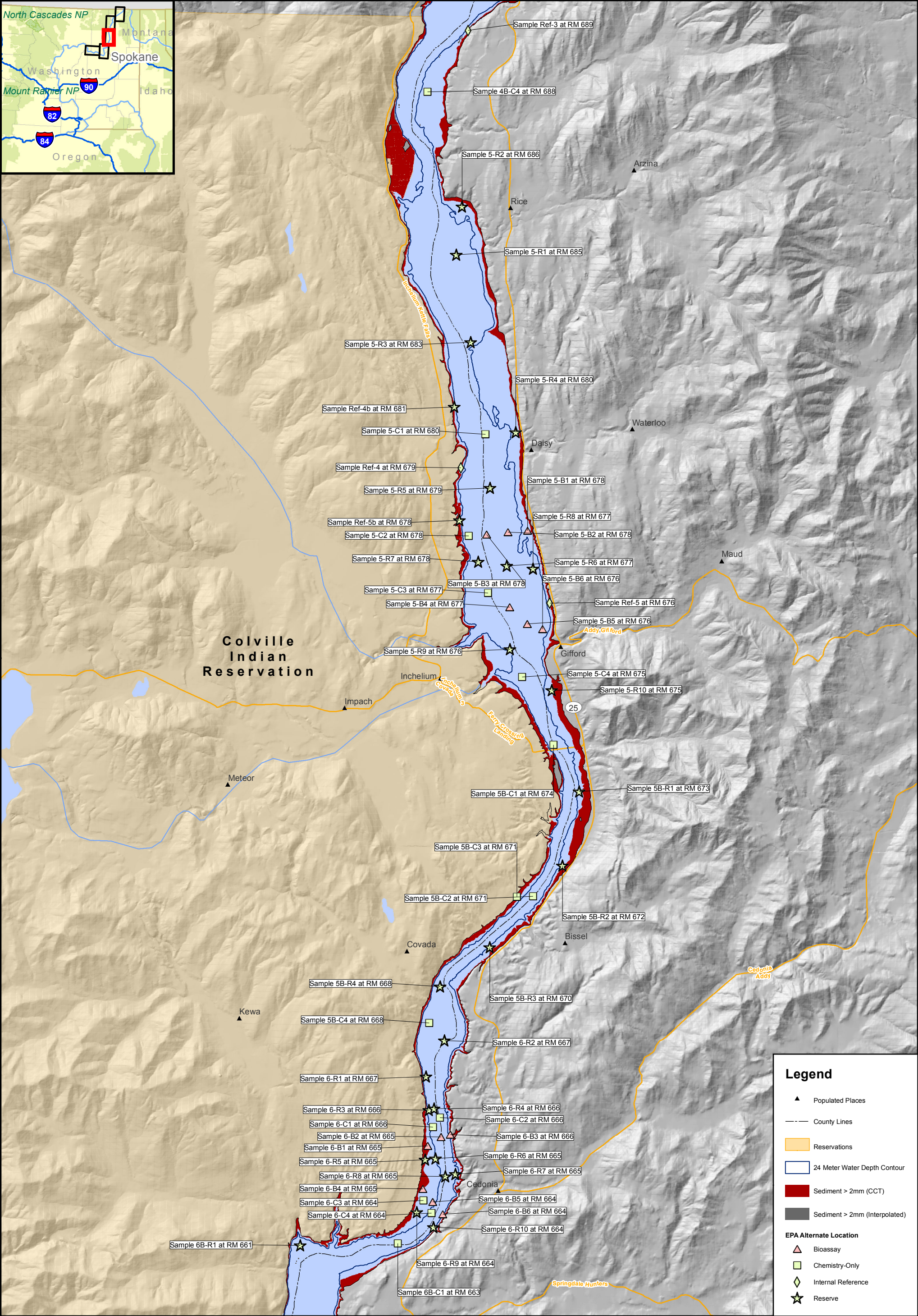




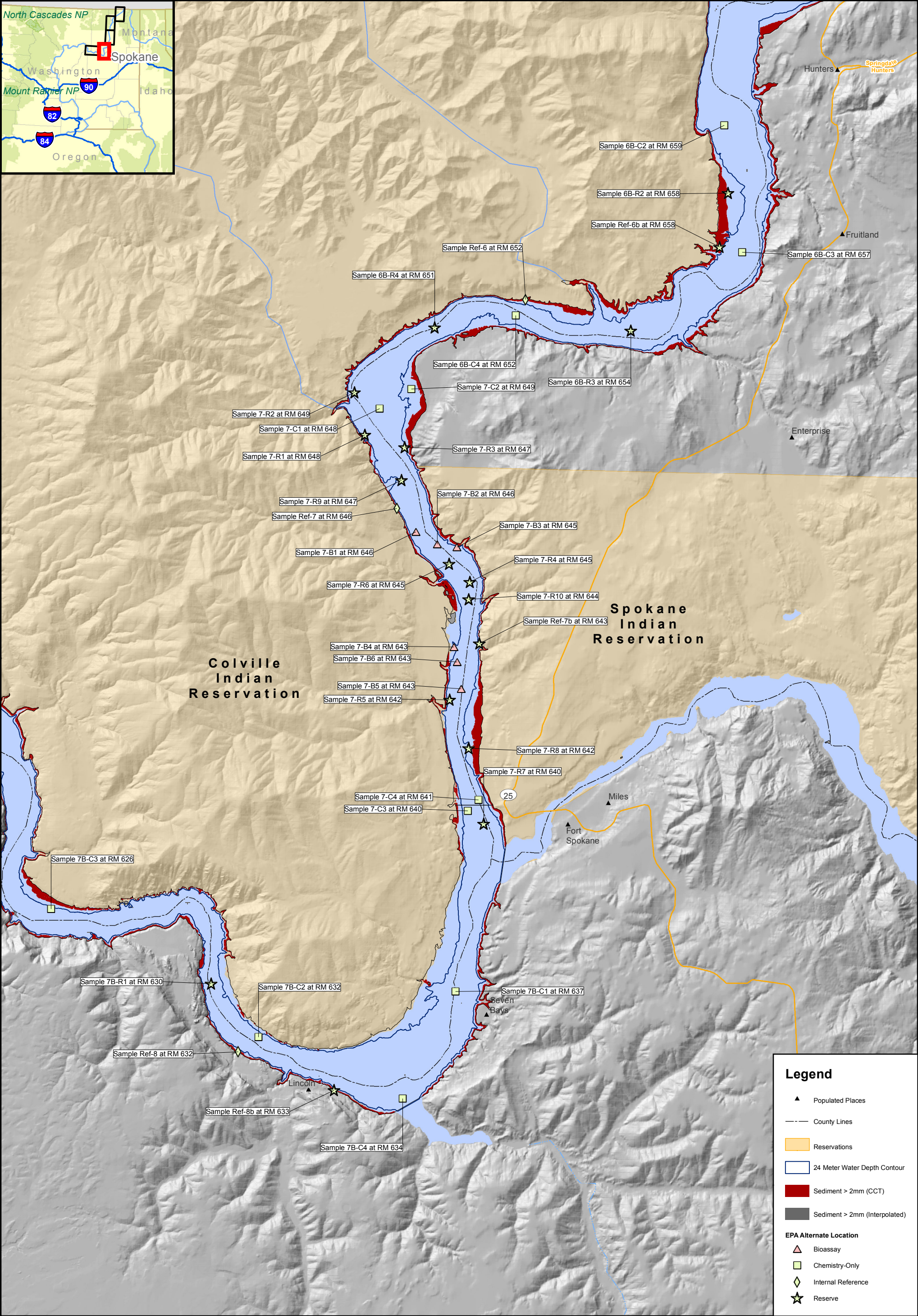




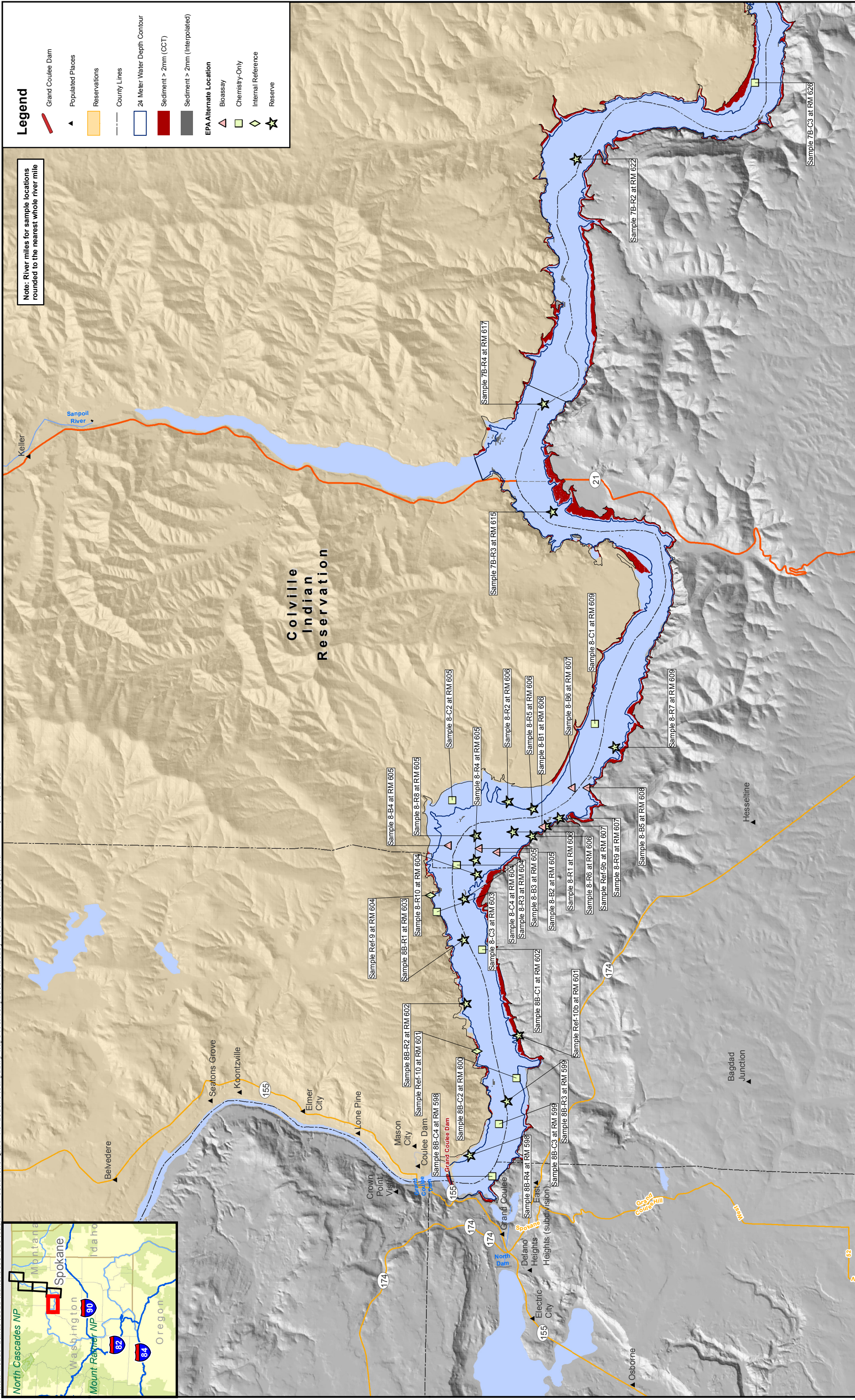




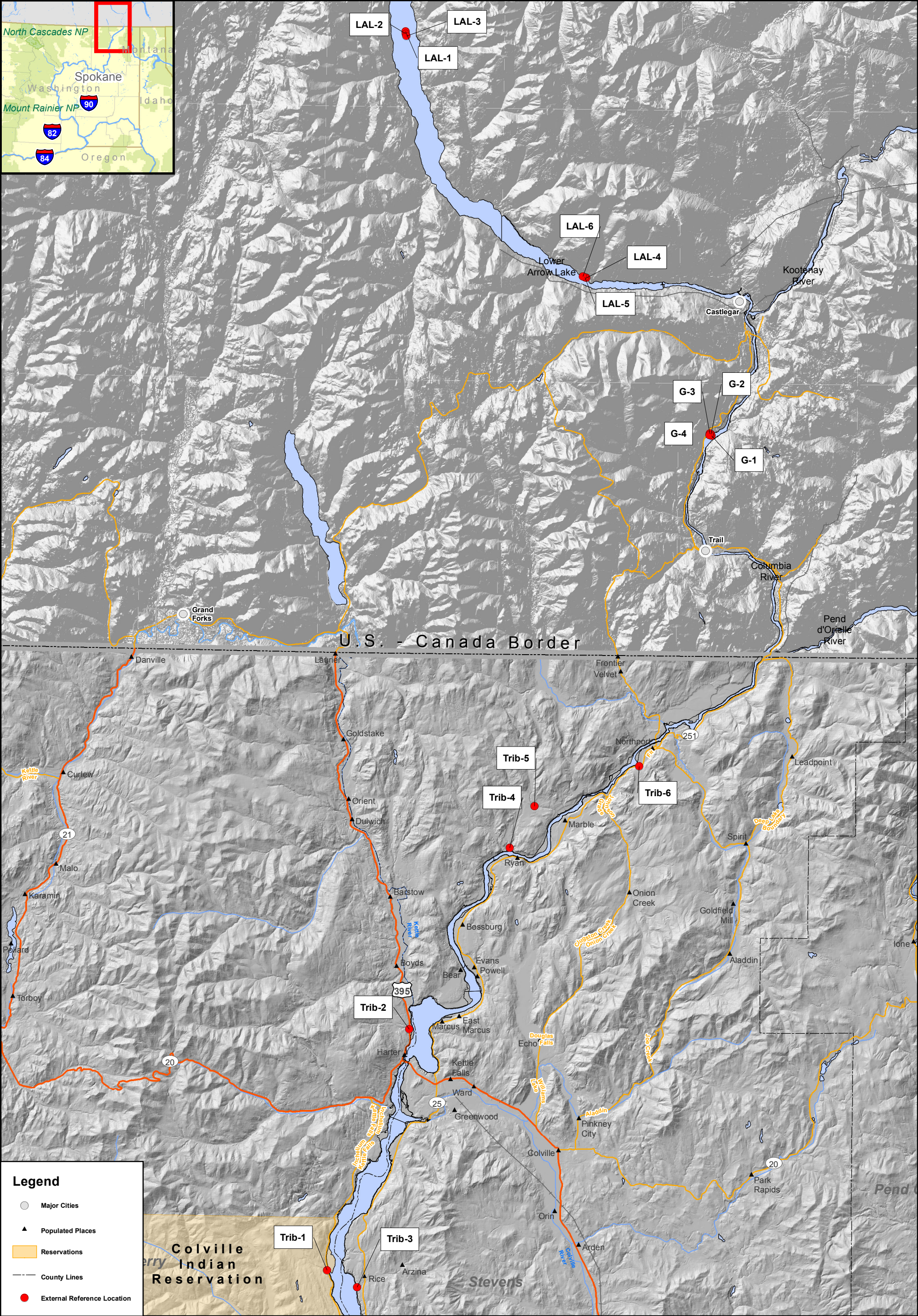




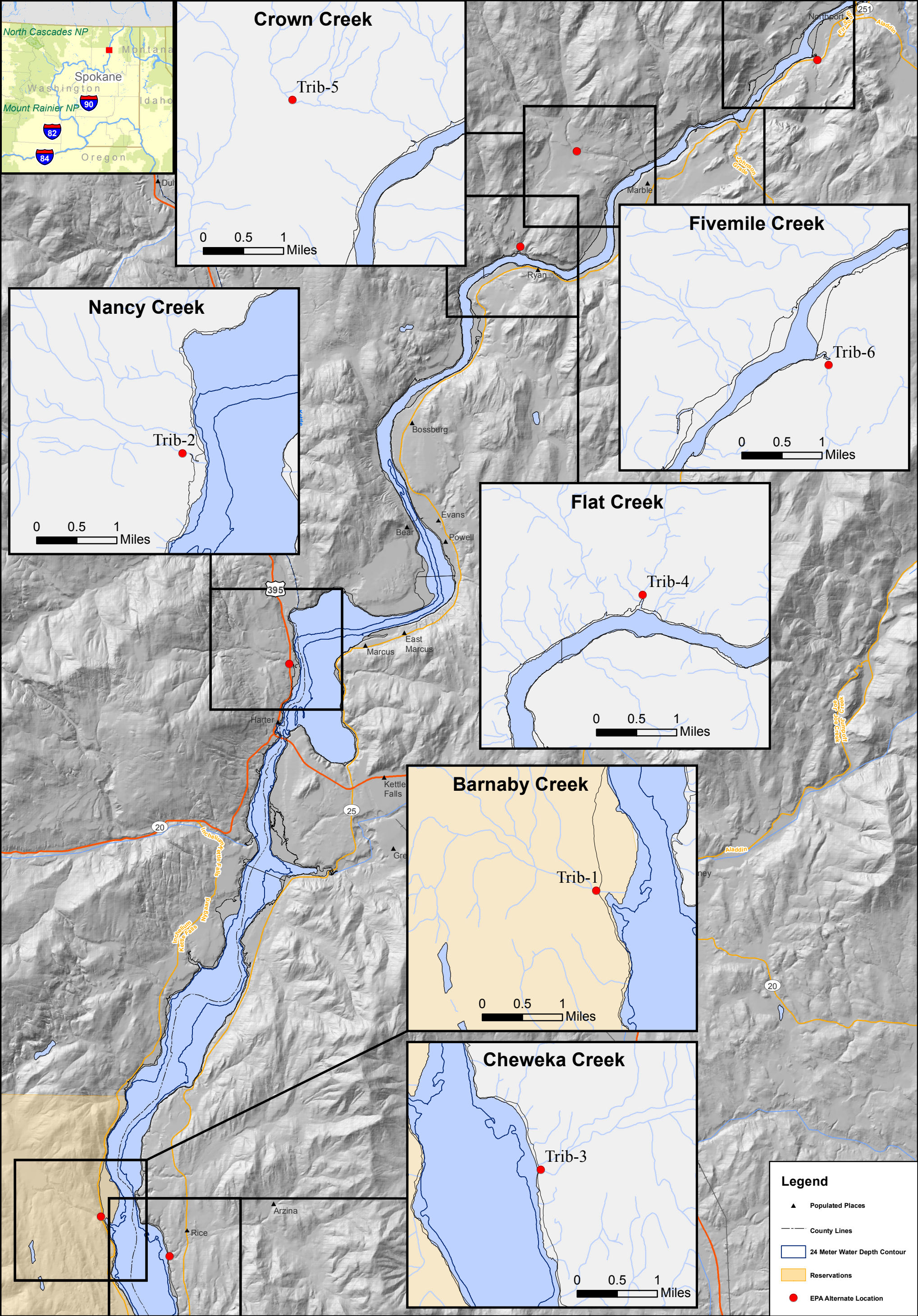




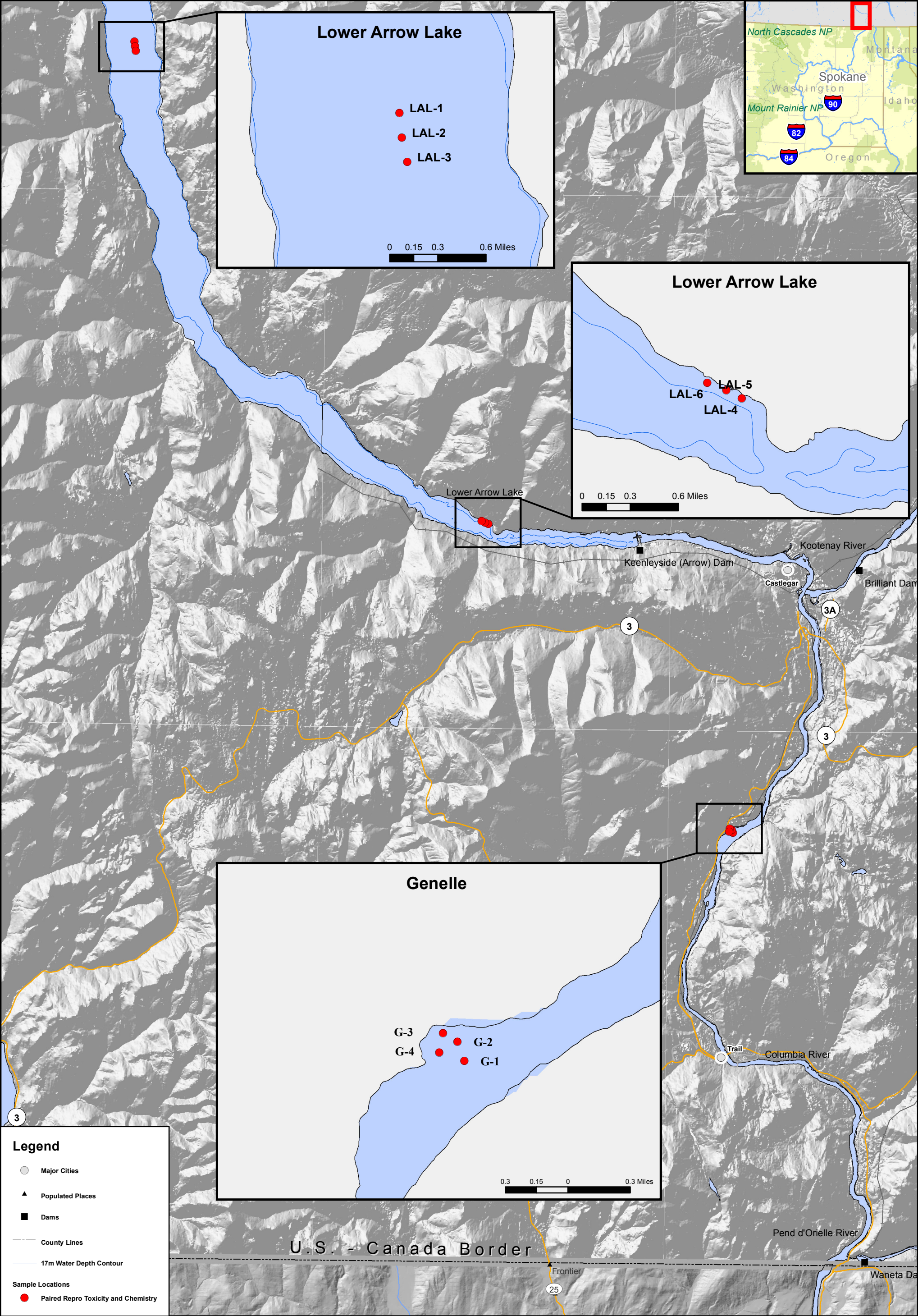














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Table A4-1. Technical Team Task Member Information

Name	Task Role	Phone	Email
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Table A5-1. Sediment/Porewater COPCs Requiring Additional Evaluation as Determined by the SLERA

Analyte	Basis for Decision
<b>Nutrients</b>	
Ammonia	Gaps in spatial coverage
Cyanide	Gaps in spatial coverage
Nitrite-Nitrate	Gaps in spatial coverage
Phosphorous	No SEV
<b>Metals/Metalloids</b>	
Aluminum	No SEV
Antimony	Maximum Measured Concentration > SEV
Arsenic	Maximum Measured Concentration > SEV
Barium	No SEV
Beryllium	Maximum Measured Concentration > SEV
Bismuth	No SEV
Boron	No SEV
Cadmium	Maximum Measured Concentration > SEV
Calcium	No SEV
Cerium	No SEV
Cesium	No SEV
Chloride	No SEV
Chromium	Maximum Measured Concentration > SEV
Cobalt	No SEV
Copper	Maximum Measured Concentration > SEV
Dysprosium	No SEV
Erbium	No SEV
Europium	No SEV
Fluoride	No SEV
Gadolinium	No SEV
Gallium	No SEV
Germanium	No SEV
Gold	Not measured
Holmium	No SEV
Indium	Not measured
Iron	No SEV
Lanthanum	No SEV
Lead	Maximum Measured Concentration > SEV
Lithium	No SEV
Lutetium	No SEV
Magnesium	No SEV
Manganese	No SEV
Mercury	Maximum Measured Concentration > SEV
Molybdenum	No SEV
Neodymium	No SEV
Nickel	Maximum Measured Concentration > SEV
Niobium	No SEV
Potassium	No SEV
Praseodymium	No SEV
Rubidium	No SEV
Samarium	No SEV
Scandium	No SEV
Selenium	No SEV
Silicon (Silica)	No SEV
Silver	Maximum Measured Concentration > SEV
Sodium	No SEV
Strontium	No SEV
Sulfur (Sulfate)	No SEV

Table A5-1. Sediment/Porewater COPCs Requiring Additional Evaluation as Determined by the SLERA

Analyte	Basis for Decision
<b>Metals/Metalloids (continued)</b>	
Tantalum	No SEV
Tellurium	Not measured
Terbium	No SEV
Thallium	No SEV
Thorium	No SEV
Thulium	No SEV
Tin	Not measured
Titanium	No SEV
Tungsten	No SEV
Uranium	No SEV
Vanadium	No SEV
Ytterbium	No SEV
Yttrium	No SEV
Zinc	Maximum Measured Concentration > SEV
Zirconium	No SEV
<b>Dioxins/Furans</b>	
1,2,3,4,6,7,8-HpCDD	No SEV
1,2,3,4,6,7,8-HpCDF	No SEV
1,2,3,4,7,8,9-HpCDF	No SEV
1,2,3,4,7,8-HxCDD	No SEV
1,2,3,4,7,8-HxCDF	No SEV
1,2,3,6,7,8-HxCDD	No SEV
1,2,3,6,7,8-HxCDF	No SEV
1,2,3,7,8,9-HxCDD	No SEV
1,2,3,7,8,9-HxCDF	No SEV
1,2,3,7,8-PCDF	No SEV
1,2,3,7,8-PCDD	No SEV
2,3,4,6,7,8-HxCDF	No SEV
2,3,4,7,8-PCDF	No SEV
2,3,7,8-TCDD	No SEV
2,3,7,8-TCDF	No SEV
Octachlorodibenzodioxin	No SEV
Octachlorodibenzofuran	No SEV
<b>TCDD TEQ</b>	Maximum Measured Concentration > SEV
<b>PBDEs</b>	
<b>Total PBDEs</b>	Not measured
<b>Pesticides</b>	
2,4'-DDD	Total DDT and Metabolites $\geq$ SEV
4,4'-DDD	Total DDT and Metabolites $\geq$ SEV
<b>Total DDD</b>	Total DDT and Metabolites $\geq$ SEV
2,4'-DDE	Total DDT and Metabolites $\geq$ SEV
4,4'-DDE	Total DDT and Metabolites $\geq$ SEV
<b>Total DDE</b>	Total DDT and Metabolites $\geq$ SEV
2,4'-DDT	Total DDT and Metabolites $\geq$ SEV
4,4'-DDT	Total DDT and Metabolites $\geq$ SEV
<b>Total DDT</b>	Total DDT and Metabolites $\geq$ SEV
<b>Total DDx</b>	Total DDT and Metabolites $\geq$ SEV
Atrazine	No SEV
alpha-BHC	No SEV
beta-BHC	No SEV
Endrin aldehyde	No SEV
Hexachlorobenzene	No SEV
Methoxychlor	Maximum Measured Concentration > SEV



Table A5-1. Sediment/Porewater COPCs Requiring Additional Evaluation as Determined by the SLERA

Analyte	Basis for Decision
<b>SVOCs</b>	
2,2'-oxybis(1-Chloropropane)	No SEV
2,4,5-Trichlorophenol	No SEV
2,4,6-Trichlorophenol	No SEV
2,4-Dichlorophenol	No SEV
2,4-Dimethylphenol	No SEV
2,4-Dinitrophenol	No SEV
2,4-Dinitrotoluene	No SEV
2,6-Dinitrotoluene	No SEV
2-Chloronaphthalene	No SEV
2-Chlorophenol	No SEV
2-Methylphenol (o-cresol)	No SEV
2-Nitroaniline	No SEV
2-Nitrophenol	No SEV
3,3'-Dichlorobenzidine	No SEV
3-Nitroaniline	No SEV
4,6-Dinitro-2-methylphenol	No SEV
4-Chloro-3-methylphenol	No SEV
4-Chloroaniline	No SEV
4-Chlorophenyl-phenyl ether	No SEV
4-Nitroaniline	No SEV
4-Nitrophenol	No SEV
Acetophenone	No SEV
Benzaldehyde	No SEV
Benzyl alcohol	No SEV
bis(2-Chloroethoxy)methane	No SEV
Bis(2-chloroethyl)ether	No SEV
Caprolactam	No SEV
Dimethyl phthalate	All Detection Limit > SEV
Di-n-octylphthalate	All Detection Limit > SEV
Hexachlorocyclopentadiene	No SEV
Isophorone	No SEV
Nitrobenzene	No SEV
N-Nitrosodi-n-propylamine	No SEV
N-Nitrosodiphenylamine	No SEV
Pentachlorophenol	No SEV
Phenol	No SEV

**Notes:**

SEV = screening ecological value

Table A7-1. Number of Sample Locations

Sample Type		Analyses	EPA Alternative Sampling Locations	
			Primary	Reserve
Site		Chemistry & Bioassay	48	114
		Chemistry Only	66	
	Internal Reference	Chemistry & Bioassay	10	10
Reference	Tributary	Chemistry & Bioassay	6	0
	Upstream	Chemistry & Bioassay	10	0
<i>Total</i>			<i>140</i>	<i>124</i>

Table A7-2. Analytes and Methods

Analytes	Sample Preparation		Quantitative Analysis		MDL/MRL Detection Limit
	Protocol	Procedure	Protocol	Procedure	
<b>Porewater Samples</b>					
Dissolved TAL metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), nickel (Ni), selenium (Se), silver (Ag), thallium (Tl), vanadium (V), and zinc (Zn)	EPA CLP	Acid Digestion	EPA 6020A	ICP/MS	See Footnote B1
Dissolved TAL metals: calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), and sodium (Na)	EPA CLP	Acid Digestion	EPA 6010C	ICP/AES	See Footnote B2
TOC and DOC	--	--	EPA 9060A	--	(0.07/0.5) mg/L
pH	--	--	SM 4500 H+ B	Electrometric	0.1 unit
Alkalinity as CaCO <sub>3</sub>	--	--	SM 2320 B	Titration	(3/9) mg/L as CaCO <sub>3</sub>
Hardness as CaCO <sub>3</sub>	--	--	SM 2340C	Calculated	(0.07/0.4) mg/L as CaCO <sub>3</sub>
Chloride, sulfate	--	--	EPA 300	Ion Chromatography	See Footnote A
<b>Sediment Samples</b>					
TAL metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), nickel (Ni), selenium (Se), silver (Ag), thallium (Tl), vanadium (V), and zinc (Zn)	EPA 3050B	Acid Digestion	EPA 6020A	ICP/MS	See Footnote B1
TAL metals: calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), and sodium (Na)	EPA 3050B	Acid Digestion	EPA 6010C	ICP/AES	See Footnote B2
Mercury (total)	EPA 7471B	Acid Digestion	EPA 7471B	Cold Vapor AA	(0.002/0.02) mg/kg
AVS/SEM	USEPA (1991)	NA	EPA 6010C/AVS-SEM	ICP/AES	See Footnote C
TOC	NA	NA	ASTM D4129-05	Coulometric	(0.02/0.05)%
pH	NA	NA	EPA 9045D	Electrometric	0.1 unit
Grain Size	NA	NA	ASTM D422	Gravimetric	NA
<b>Notes:</b>					
AA = atomic absorption					
AES = atomic emission spectrometry					
ASTM = American Society for Testing and Materials					
AVS = acid volatile sulfides					
AVS/SEM = acid volatile sulfides/simultaneously extractable metals					
CaCO <sub>3</sub> = calcium carbonate					
CLP = Contract Laboratory Program					
DOC = dissolved organic carbon					
EPA = U.S. Environmental Protection Agency					
ICP = inductively coupled plasma					
MDL = method detection limit					
MRL = method reporting limit					
MS = mass spectrometry					
NA = not applicable					
SEM = simultaneously extractable metals					
SM = Standard Methods for the Examination of Water and Wastewater					
TAL = target analyte list					
TOC = total organic carbon					
<b>B1. Reporting Limits for EPA Method 6020A (MDL   MRL)</b>					
<b>Porewater</b>					
Al = 0.3   2 µg/L					
Sb = 0.02   0.05 µg/L					
As = 0.1   0.5 µg/L					
Ba = 0.02   0.05 µg/L					
Be = 0.006   0.02 µg/L					
Cd = 0.005   0.02 µg/L					
Cr = 0.04   0.2 µg/L					
Co = 0.006   0.02 µg/L					
Cu = 0.02   0.1 µg/L					
Pb = 0.005   0.02 µg/L					
Mn = 0.006   0.05 µg/L					
Ni = 0.03   0.2 µg/L					
Se = 0.3   1 µg/L					
Ag = 0.004   0.02 µg/L					
Tl = 0.005   0.02 µg/L					
V = 0.03   0.2 µg/L					
Zn = 0.2   0.5 µg/L					
<b>Sediment</b>					
Al = 0.4   2 mg/kg dw					
Sb = 0.02   0.05 mg/kg dw					
As = 0.06   0.5 mg/kg dw					
Ba = 0.005   0.05 mg/kg dw					
Be = 0.003   0.02 mg/kg dw					
Cd = 0.004   0.02 mg/kg dw					
Cr = 0.03   0.2 mg/kg dw					
Co = 0.003   0.02 mg/kg dw					
Cu = 0.08   0.1 mg/kg dw					
Pb = 0.009   0.05 mg/kg dw					
Mn = 0.03   0.05 mg/kg dw					
Ni = 0.03   0.2 mg/kg dw					
Se = 0.2   1 mg/kg dw					
Ag = 0.008   0.02 mg/kg dw					
Tl = 0.003   0.02 mg/kg dw					
V = 0.02   0.2 mg/kg dw					
Zn = 0.2   0.5 mg/kg dw					

Table A7-3. Derivation of Analytical Concentration Goals and Proposed Laboratory Reporting and Detection Limits Based on Ecological Screening Criteria and Available Data for the Site

Analyte/Parameter	Ecological Screening Criteria					Porewater		Sediment	
	Chronic EPA NAWQC (µg/L)	Ecology Chronic WQS (µg/L)	CCT Aquatic Life Chronic Criteria (µg/L)	STI Aquatic Life Chronic Criteria (µg/L)		MDL (µg/L)	MRL (µg/L) <sup>a</sup>	Toxicity Benchmark Values (mg/kg dw) <sup>b</sup>	MRL (mg/kg dw) <sup>a</sup>
<b>Conventional Parameters</b>									
Alkalinity	NA	NA	NA	NA		3,000	9,000	NA	NA
DOC	NA	NA	NA	NA		NA	NA	NA	NA
TOC	NA	NA	NA	NA		NA	NA	NA	0.05 %
Hardness	NA	NA	NA	NA		800	2,000	NA	NA
pH	NA	NA	NA	NA		NA	NA	NA	0.5 (unitless)
<b>Cations/Anions</b>									
Calcium	NA	NA	NA	NA		9	50	NA	10
Chloride	230,000	230,000	230,000	230,000		30	200	NA	NA
Magnesium	NA	NA	NA	NA		0.4	20	NA	4
Potassium	NA	NA	NA	NA		40	400	NA	80
Sodium	NA	NA	NA	NA		20	200	NA	40
Sulfate	NA	NA	NA	NA		10	200	NA	NA
<b>Metals and Metalloids<sup>c</sup></b>									
Aluminum	87	NA	87	87		0.3	2	NA	2
Antimony	NA	NA	NA	NA		0.02	0.05	NA	0.05
Arsenic	150	190	150	150		0.1	0.5	9.79	0.5
Barium	NA	NA	NA	NA		0.02	0.05	NA	0.05
Beryllium	NA	NA	NA	NA		0.006	0.02	NA	0.02
Cadmium	0.25	0.77 <sup>d</sup>	0.19 <sup>d</sup>	0.77 <sup>d</sup>		0.005	0.02	0.99	0.02
Chromium	74	128 <sup>d</sup>	53 <sup>d</sup>	53		0.04	0.2	43.4	0.2
Cobalt	NA	NA	NA	NA		0.006	0.02	NA	0.02
Copper	9.0	8.1 <sup>d</sup>	6.4 <sup>d</sup>	6.4		0.02	0.1	31.6	0.1
Iron	1,000	NA	1,000	1,000		3	20	NA	4
Lead	2.5	1.6 <sup>d</sup>	1.6 <sup>d</sup>	1.6		0.005	0.02	35.8	0.05
Manganese	NA	NA	NA	NA		0.006	0.05	NA	0.05

Table A7-3. Derivation of Analytical Concentration Goals and Proposed Laboratory Reporting and Detection Limits Based on Ecological Screening Criteria and Available Data for the Site

Analyte/Parameter	Ecological Screening Criteria					Porewater		Sediment	
	Chronic EPA NAWQC (µg/L)	Ecology Chronic WQS (µg/L)	CCT Aquatic Life Chronic Criteria (µg/L)	STI Aquatic Life Chronic Criteria (µg/L)		MDL (µg/L)	MRL (µg/L) <sup>a</sup>	Toxicity Benchmark Values (mg/kg dw) <sup>b</sup>	MRL (mg/kg dw) <sup>a</sup>
<b>Metals and Metalloids<sup>c</sup> (continued)</b>									
Mercury	0.80	0.012	0.80	0.012		NA	NA	0.18	0.02
Nickel	52 <sup>d</sup>	112 <sup>d</sup>	37 <sup>d</sup>	37 <sup>d</sup>		0.03	0.2	22.7	0.2
Selenium	5.0	20	5.0	5.0		0.3	1	NA	1
Silver	1.6 <sup>d</sup>	1.7 <sup>d</sup>	1.6 <sup>d</sup>	1.7 <sup>d</sup>		0.004	0.02	NA	0.02
Thallium	NA	NA	NA	NA		0.005	0.02	NA	0.02
Vanadium	NA	NA	NA	NA		0.03	0.2	NA	2
Zinc	120	74	84	74		0.2	0.5	121	0.5

**Notes:**<sup>a</sup> Non-detects will be reported to the MDL. Values between the MDL and the MRL will be estimated (i.e., "J" qualified).<sup>b</sup> Based on consensus-based threshold effect concentrations (TECs.) Source: MacDonald et al. (2000).<sup>c</sup> Water samples are analyzed for dissolved metals and sediment samples are analyzed for total metals.<sup>d</sup> Criteria are hardness or pH dependent and are calculated using the means of those parameters from the Ecology (2006) surface water data.

Mean hardness = 66.89 mg/L (range 58.3 to 77.3 mg/L), Mean pH = 8.11 standard units, Mean temperature = 9.5°C.

NAWQC = national ambient water quality criteria

CCT = Colville Confederated Tribes

DOC = dissolved organic carbon

dw = dry weight

MDL = method detection limit

MRL = method reporting limit

NA = not applicable

STI = Spokane Tribe of Indians

TEC = threshold exposure concentrations

TOC = total organic carbon

WQS = water quality standard

Table B1-1. Sampling Locations

River Mile	CSM Unit	X_UTM_11N	Y_UTM_11N	Latitude	Longitude
744	CSM Unit 1	453509.4201	5427422.5025	48.9980	-117.6356
743	CSM Unit 1	453049.2160	5425706.7652	48.9825	-117.6417
742	CSM Unit 1	451648.4545	5424098.8892	48.9679	-117.6606
740	CSM Unit 1	449383.7152	5422536.5077	48.9537	-117.6914
739	CSM Unit 1	447418.4558	5421563.1309	48.9448	-117.7181
739	CSM Unit 1	447786.7914	5421919.1414	48.9480	-117.7131
738	CSM Unit 1	446702.4589	5420858.9288	48.9384	-117.7278
738	CSM Unit 1	446805.9822	5420459.5044	48.9348	-117.7263
737	CSM Unit 1	445699.4516	5420996.5968	48.9395	-117.7415
737	CSM Unit 1	446362.5845	5421153.1449	48.9410	-117.7325
735	CSM Unit 1	443177.8505	5418969.8971	48.9211	-117.7756
734	CSM Unit 1	442907.6141	5418852.1021	48.9200	-117.7793
734	CSM Unit 1	442467.0883	5418819.2320	48.9196	-117.7853
733	CSM Unit 1	441090.2453	5417255.8861	48.9054	-117.8039
733	CSM Unit 1	441604.8343	5418331.8254	48.9152	-117.7970
732	CSM Unit 1	440379.5707	5416787.8301	48.9012	-117.8135
731	CSM Unit 1	438638.5012	5415121.2071	48.8860	-117.8370
731	CSM Unit 1	439803.8784	5415827.6896	48.8925	-117.8212
731	CSM Unit 1	439332.2297	5416009.6655	48.8941	-117.8277
730	CSM Unit 1	438161.3082	5414310.6777	48.8787	-117.8434
730	CSM Unit 1	438059.8053	5414097.9036	48.8767	-117.8448
730	CSM Unit 1	437662.4861	5414117.1880	48.8769	-117.8502
729	CSM Unit 1	436880.8238	5413705.7781	48.8731	-117.8608
729	CSM Unit 1	436540.9523	5413587.1195	48.8720	-117.8654
728	CSM Unit 1	435609.5237	5413195.8161	48.8684	-117.8780
727	CSM Unit 1	434669.4030	5412226.0305	48.8596	-117.8907
727	CSM Unit 1	434190.3665	5411916.3190	48.8567	-117.8972
726	CSM Unit 1	433680.1512	5411584.2567	48.8537	-117.9041
725	CSM Unit 1	432384.0675	5410533.4624	48.8441	-117.9216
724	CSM Unit 1	432191.0169	5408515.6151	48.8259	-117.9239
724	CSM Unit 1	431675.6534	5408577.7051	48.8264	-117.9309
724	CSM Unit 1	431407.7056	5407978.5225	48.8210	-117.9345
724	CSM Unit 1	431999.6766	5407758.1616	48.8191	-117.9264
723	CSM Unit 1	431061.2423	5407494.7208	48.8166	-117.9391
723	CSM Unit 1	430516.6068	5407063.5406	48.8127	-117.9464
723	CSM Unit 1	430251.0952	5406968.7694	48.8118	-117.9500
722	CSM Unit 1	429259.6594	5407490.5539	48.8164	-117.9636
721	CSM Unit 1	428055.4950	5407623.6644	48.8174	-117.9801
720	CSM Unit 1	426962.7318	5407475.5179	48.8160	-117.9949
719	CSM Unit 1	426117.5261	5404740.7728	48.7913	-118.0059
715	CSM Unit 1	422930.3481	5401213.9989	48.7592	-118.0487
714	CSM Unit 1	422143.2689	5398707.5541	48.7365	-118.0589
710	CSM Unit 2	424498.5577	5393545.3791	48.6904	-118.0259
710	CSM Unit 2	424141.7407	5392332.5779	48.6794	-118.0305
708	CSM Unit 2	422307.5206	5391562.8467	48.6723	-118.0553
707	CSM Unit 2	420548.2110	5391412.7358	48.6707	-118.0792
707	CSM Unit 2	418339.8305	5392015.8116	48.6758	-118.1093
706	CSM Unit 2	418971.9838	5390738.2203	48.6644	-118.1004
706	CSM Unit 2	419432.7220	5390220.0593	48.6598	-118.0941
705	CSM Unit 2	418628.2125	5388994.1163	48.6487	-118.1048
705	CSM Unit 2	419070.5788	5388803.6176	48.6470	-118.0987
704	CSM Unit 2	418880.1322	5387654.2160	48.6367	-118.1011
704	CSM Unit 2	420010.3993	5387622.5715	48.6365	-118.0857
703	CSM Unit 2	419944.9095	5386148.0589	48.6233	-118.0863
701	CSM Unit 2	416956.7457	5384334.9785	48.6066	-118.1265
699	CSM Unit 2	416050.4669	5380836.1877	48.5750	-118.1381

Table B1-1. Sampling Locations

River Mile	CSM Unit	X_UTM_11N	Y_UTM_11N	Latitude	Longitude
698	CSM Unit 3	416735.0144	5379566.6837	48.5637	-118.1286
696	CSM Unit 3	414919.3797	5376770.9835	48.5383	-118.1526
692	CSM Unit 3	413016.4130	5373254.3451	48.5064	-118.1777
689	CSM Unit 3	412113.9581	5370135.7474	48.4782	-118.1892
688	CSM Unit 3	410840.2606	5368231.4983	48.4609	-118.2060
680	CSM Unit 3	412646.8818	5357580.3021	48.3653	-118.1794
679	CSM Unit 3	411884.2490	5356534.9157	48.3558	-118.1895
678	CSM Unit 3	413961.7518	5354583.3221	48.3386	-118.1610
678	CSM Unit 3	413349.6141	5354533.6893	48.3380	-118.1693
678	CSM Unit 3	412683.5026	5354463.5628	48.3373	-118.1782
678	CSM Unit 3	412129.8358	5354418.5881	48.3368	-118.1857
677	CSM Unit 3	413398.2903	5352205.2719	48.3171	-118.1681
677	CSM Unit 3	412726.9162	5352643.6036	48.3210	-118.1773
676	CSM Unit 3	413948.6113	5351681.2164	48.3125	-118.1606
676	CSM Unit 3	414422.9844	5351520.9095	48.3111	-118.1542
676	CSM Unit 3	414649.1196	5352332.8588	48.3184	-118.1513
675	CSM Unit 3	413784.3845	5350035.8974	48.2976	-118.1625
674	CSM Unit 3	414765.0562	5347924.3964	48.2788	-118.1488
671	CSM Unit 3	413628.0184	5343195.2220	48.2361	-118.1632
671	CSM Unit 3	414124.0527	5343215.7328	48.2363	-118.1565
668	CSM Unit 3	410901.1046	5339274.8928	48.2005	-118.1991
666	CSM Unit 3	411556.0854	5335806.6497	48.1693	-118.1896
666	CSM Unit 3	411018.7702	5336033.4157	48.1713	-118.1968
666	CSM Unit 3	411227.3782	5336335.0334	48.1741	-118.1941
665	CSM Unit 3	410854.7068	5335454.2515	48.1661	-118.1989
665	CSM Unit 3	410705.3603	5334113.1373	48.1540	-118.2006
665	CSM Unit 3	411268.5670	5335730.8443	48.1686	-118.1934
664	CSM Unit 3	411008.6049	5333713.5271	48.1504	-118.1965
664	CSM Unit 3	411317.0992	5333324.1709	48.1470	-118.1923
664	CSM Unit 3	410720.0991	5333756.5288	48.1508	-118.2004
664	CSM Unit 3	410970.5675	5333359.2566	48.1472	-118.1969
663	CSM Unit 3	409919.5526	5332421.6511	48.1387	-118.2108
659	CSM Unit 3	407026.5480	5328246.9336	48.1007	-118.2488
657	CSM Unit 3	407578.6532	5324298.4494	48.0653	-118.2405
652	CSM Unit 3	400540.3906	5322333.7476	48.0465	-118.3345
652	CSM Unit 3	400847.7260	5322829.8370	48.0510	-118.3305
649	CSM Unit 3	397295.6854	5320051.8665	48.0255	-118.3775
648	CSM Unit 3	396304.1652	5319441.8134	48.0198	-118.3907
646	CSM Unit 3	397434.7901	5315605.9057	47.9855	-118.3746
646	CSM Unit 3	398099.5200	5315221.9882	47.9822	-118.3656
646	CSM Unit 3	396847.3055	5316334.6183	47.9920	-118.3826
645	CSM Unit 3	398713.0791	5315131.1322	47.9815	-118.3573
643	CSM Unit 3	398618.1482	5312027.9462	47.9535	-118.3579
643	CSM Unit 3	398839.9041	5310727.6951	47.9419	-118.3546
643	CSM Unit 3	398714.8503	5311566.1192	47.9494	-118.3565
641	CSM Unit 3	399383.8076	5307273.0598	47.9109	-118.3465
640	CSM Unit 3	399053.1499	5306933.8442	47.9078	-118.3509
637	CSM Unit 3	398667.3963	5301315.6713	47.8572	-118.3547
634	CSM Unit 3	397025.1674	5297988.6596	47.8270	-118.3759
632	CSM Unit 3	392537.3457	5299898.6690	47.8434	-118.4363
632	CSM Unit 3	391906.6828	5299446.3188	47.8393	-118.4446
626	CSM Unit 3	386089.6203	5303883.2933	47.8782	-118.5235
609	CSM Unit 3	366204.1236	5308848.2973	47.9190	-118.7908
608	CSM Unit 3	364224.4362	5309115.9473	47.9210	-118.8174
607	CSM Unit 3	364223.8196	5309575.7299	47.9251	-118.8176
606	CSM Unit 3	363009.4477	5310489.9501	47.9331	-118.8341

Table B1-1. Sampling Locations

River Mile	CSM Unit	X_UTM_11N	Y_UTM_11N	Latitude	Longitude
605	CSM Unit 3	362219.5509	5311913.6540	47.9457	-118.8451
605	CSM Unit 3	362335.6817	5312464.3123	47.9507	-118.8438
605	CSM Unit 3	362427.0537	5313415.7405	47.9593	-118.8428
605	CSM Unit 3	363836.2186	5313271.5550	47.9583	-118.8239
604	CSM Unit 3	361825.4727	5313132.1417	47.9566	-118.8508
604	CSM Unit 3	360887.9216	5313945.7006	47.9637	-118.8636
603	CSM Unit 3	360370.7016	5313754.7389	47.9619	-118.8705
602	CSM Unit 3	359207.2189	5312342.1436	47.9489	-118.8856
601	CSM Unit 3	356060.9582	5312511.8486	47.9497	-118.9278
600	CSM Unit 3	355217.8178	5311280.4868	47.9385	-118.9386
599	CSM Unit 3	353792.3763	5311819.0023	47.9430	-118.9579
598	CSM Unit 3	352173.8334	5312027.8193	47.9445	-118.9796
<b>External Reference Locations</b>					
Trib-1	Barnaby Creek	409599.0882	5365221.8770	48.4337	-118.2222
Trib-2	Nancy Creek	417960.0043	5389749.2880	48.6554	-118.1140
Trib-3	Cheweka Creek	412656.5780	5363476.2147	48.4184	-118.1805
Trib-4	Flat Creek	428210.3396	5408246.6044	48.8231	-117.9781
Trib-5	Crown Creek	430719.1785	5412475.2448	48.8614	-117.9446
Trib-6	Fivemile Creek	441398.6667	5416524.0973	48.8989	-117.7996
Lower Arrow Lake		417590.4138	5491377.5174	49.5694	-118.1398
Lower Arrow Lake		417614.5377	5491131.9909	49.5672	-118.1394
Lower Arrow Lake		417669.4369	5490887.5742	49.5650	-118.1386
Lower Arrow Lake		435667.1209	5466414.5085	49.3471	-117.8857
Lower Arrow Lake		435858.3129	5466340.1099	49.3464	-117.8831
Lower Arrow Lake		436014.4621	5466259.1383	49.3457	-117.8809
Genelle		448590.9184	5450405.5787	49.2043	-117.7058
Genelle		448699.7059	5450340.7110	49.2037	-117.7043
Genelle		448560.9976	5450257.5264	49.2030	-117.7061
Genelle		448752.8572	5450192.3689	49.2024	-117.7035
<b>Reserve Locations</b>					
744	CSM Unit 1	453467.0850	5427258.8783	48.9965	-117.6362
744	CSM Unit 1	453182.7745	5426533.4896	48.9899	-117.6400
744	CSM Unit 1	453588.2571	5427558.5769	48.9992	-117.6345
743	CSM Unit 1	453044.9424	5425055.4056	48.9766	-117.6417
742	CSM Unit 1	452591.6512	5424701.4318	48.9734	-117.6478
741	CSM Unit 1	451031.6871	5423758.5958	48.9648	-117.6690
739	CSM Unit 1	448355.4421	5422309.9013	48.9516	-117.7054
737	CSM Unit 1	363791.7652	5311551.4066	47.9428	-118.8240
737	CSM Unit 1	445899.6744	5421131.3897	48.9407	-117.7388
737	CSM Unit 1	446122.3203	5420985.1424	48.9394	-117.7357
735	CSM Unit 1	442963.6188	5419186.2676	48.9230	-117.7786
735	CSM Unit 1	443093.4939	5418969.8631	48.9210	-117.7768
735	CSM Unit 1	443543.8270	5419245.9786	48.9236	-117.7707
734	CSM Unit 1	442815.2564	5418839.6755	48.9199	-117.7806
732	CSM Unit 1	441061.5339	5417081.8569	48.9039	-117.8043
732	CSM Unit 1	440549.5791	5417293.9410	48.9057	-117.8113
732	CSM Unit 1	440012.8552	5416681.7387	48.9002	-117.8185
731	CSM Unit 1	439370.7699	5415630.5437	48.8907	-117.8271
731	CSM Unit 1	438796.5388	5415696.0756	48.8912	-117.8350
731	CSM Unit 1	438903.3279	5415215.3541	48.8869	-117.8334
730	CSM Unit 1	438384.6746	5414794.8399	48.8830	-117.8404
730	CSM Unit 1	438147.4244	5414464.0447	48.8800	-117.8436
729	CSM Unit 1	437139.6506	5413801.3798	48.8740	-117.8573
729	CSM Unit 1	436564.8183	5413686.5724	48.8729	-117.8651
728	CSM Unit 1	435504.0364	5412975.9687	48.8664	-117.8794
728	CSM Unit 1	435831.0622	5413561.7065	48.8717	-117.8751



Table B1-1. Sampling Locations

River Mile	CSM Unit	X_UTM_11N	Y_UTM_11N	Latitude	Longitude
<b>Reserve Locations (continued)</b>					
727	CSM Unit 1	433879.2918	5411733.6064	48.8550	-117.9014
726	CSM Unit 1	432851.5242	5411157.8576	48.8498	-117.9153
726	CSM Unit 1	433366.8018	5411453.6148	48.8525	-117.9083
725	CSM Unit 1	432015.0278	5409798.1401	48.8374	-117.9265
725	CSM Unit 1	432291.9754	5409134.4831	48.8315	-117.9226
724	CSM Unit 1	431500.1156	5408365.3311	48.8245	-117.9333
723	CSM Unit 1	431574.9290	5407627.9344	48.8179	-117.9321
723	CSM Unit 1	430826.6285	5407387.8550	48.8156	-117.9423
722	CSM Unit 1	430277.8522	5407169.9098	48.8136	-117.9497
722	CSM Unit 1	429445.7016	5407239.6105	48.8141	-117.9610
721	CSM Unit 1	428466.8141	5407797.1528	48.8190	-117.9745
721	CSM Unit 1	427574.4198	5407796.8690	48.8189	-117.9866
720	CSM Unit 1	426267.6107	5406857.8863	48.8103	-118.0043
718	CSM Unit 1	425881.6760	5403961.6779	48.7842	-118.0090
717	CSM Unit 1	424926.1779	5402793.9814	48.7736	-118.0218
715	CSM Unit 1	422213.0046	5400342.1397	48.7512	-118.0582
712	CSM Unit 1	423883.4961	5395557.8842	48.7084	-118.0346
711	CSM Unit 1	424342.0331	5394268.8047	48.6969	-118.0282
709	CSM Unit 2	423074.9214	5391768.8503	48.6742	-118.0449
708	CSM Unit 2	421223.0965	5391914.9176	48.6753	-118.0701
707	CSM Unit 2	419942.0117	5391598.5128	48.6723	-118.0874
707	CSM Unit 2	419261.0478	5391742.5653	48.6735	-118.0967
707	CSM Unit 2	420001.0260	5392779.1715	48.6829	-118.0869
705	CSM Unit 2	418993.5109	5389358.0759	48.6520	-118.0999
705	CSM Unit 2	419822.6609	5389209.6489	48.6508	-118.0886
705	CSM Unit 2	418517.0001	5390073.1658	48.6584	-118.1065
704	CSM Unit 2	419061.7584	5387607.3498	48.6363	-118.0986
703	CSM Unit 2	420521.5816	5385568.7634	48.6181	-118.0784
702	CSM Unit 2	416976.9193	5384847.9590	48.6112	-118.1264
701	CSM Unit 2	416708.7224	5384856.6594	48.6112	-118.1300
698	CSM Unit 3	416289.2542	5379288.1317	48.5611	-118.1346
697	CSM Unit 3	415116.9224	5378552.6767	48.5543	-118.1503
693	CSM Unit 3	413755.1486	5374478.6852	48.5175	-118.1679
693	CSM Unit 3	412745.7854	5374990.0535	48.5220	-118.1817
691	CSM Unit 3	413570.1954	5372295.3335	48.4978	-118.1700
686	CSM Unit 3	411920.5609	5364655.0886	48.4289	-118.1907
685	CSM Unit 3	411741.7980	5363158.6333	48.4154	-118.1928
683	CSM Unit 3	412187.3026	5360437.4712	48.3910	-118.1862
681	CSM Unit 3	411681.5116	5358429.8534	48.3729	-118.1926
680	CSM Unit 3	413586.0810	5357643.6493	48.3660	-118.1667
679	CSM Unit 3	412794.8433	5355906.5887	48.3503	-118.1770
678	CSM Unit 3	412420.6447	5353611.4682	48.3296	-118.1816
678	CSM Unit 3	411844.1665	5354906.8003	48.3412	-118.1897
677	CSM Unit 3	413310.8246	5353490.8058	48.3287	-118.1696
677	CSM Unit 3	414130.0949	5353398.1086	48.3279	-118.1585
676	CSM Unit 3	413414.0224	5350894.0804	48.3053	-118.1677
675	CSM Unit 3	414715.6921	5349616.9854	48.2940	-118.1498
673	CSM Unit 3	415561.3346	5346467.4193	48.2658	-118.1378
672	CSM Unit 3	415045.5045	5344167.9850	48.2450	-118.1443
670	CSM Unit 3	412780.9335	5341626.0134	48.2219	-118.1743
668	CSM Unit 3	411236.8950	5340403.7388	48.2107	-118.1948
667	CSM Unit 3	410799.9953	5337610.3087	48.1855	-118.2001
667	CSM Unit 3	411375.8938	5338732.3050	48.1956	-118.1926
666	CSM Unit 3	410888.9402	5336566.4881	48.1761	-118.1987
666	CSM Unit 3	411054.1045	5336613.0865	48.1765	-118.1965

Table B1-1. Sampling Locations

River Mile	CSM Unit	X_UTM_11N	Y_UTM_11N	Latitude	Longitude
<b>Reserve Locations (continued)</b>					
665	CSM Unit 3	410772.7857	5335024.3225	48.1622	-118.1999
665	CSM Unit 3	411095.7067	5335058.6293	48.1626	-118.1956
665	CSM Unit 3	411713.3021	5334577.0184	48.1583	-118.1872
665	CSM Unit 3	411402.9060	5334503.1453	48.1576	-118.1913
664	CSM Unit 3	410510.2992	5333386.1065	48.1474	-118.2031
664	CSM Unit 3	411036.2171	5332925.5305	48.1434	-118.1959
661	CSM Unit 3	406892.3536	5332357.5447	48.1377	-118.2515
658	CSM Unit 3	407143.2772	5326143.9382	48.0818	-118.2468
658	CSM Unit 3	406881.7821	5324463.8014	48.0667	-118.2499
654	CSM Unit 3	404117.6052	5321860.1674	48.0428	-118.2864
651	CSM Unit 3	398017.9043	5321965.2712	48.0428	-118.3683
649	CSM Unit 3	395521.8959	5319939.5152	48.0242	-118.4013
648	CSM Unit 3	395851.5591	5318623.1128	48.0124	-118.3965
647	CSM Unit 3	397086.0290	5318241.1340	48.0092	-118.3799
647	CSM Unit 3	396985.9692	5317216.6610	47.9999	-118.3810
645	CSM Unit 3	399113.4720	5314048.0094	47.9718	-118.3517
645	CSM Unit 3	398469.3868	5314615.9455	47.9768	-118.3605
644	CSM Unit 3	399080.6682	5313509.3701	47.9669	-118.3520
643	CSM Unit 3	399407.0937	5312138.6304	47.9547	-118.3473
642	CSM Unit 3	399075.2655	5308876.3601	47.9253	-118.3510
642	CSM Unit 3	398488.3346	5310388.8622	47.9388	-118.3592
640	CSM Unit 3	399543.1256	5306527.2679	47.9042	-118.3442
633	CSM Unit 3	394888.1888	5298252.7991	47.8290	-118.4045
630	CSM Unit 3	391064.1153	5301561.2741	47.8582	-118.4564
622	CSM Unit 3	383720.7709	5309442.3438	47.9277	-118.5566
617	CSM Unit 3	376117.8436	5310454.1125	47.9354	-118.6587
615	CSM Unit 3	372773.0327	5310180.1736	47.9323	-118.7034
609	CSM Unit 3	365474.6107	5308243.7408	47.9134	-118.8004
607	CSM Unit 3	363277.3454	5309964.2159	47.9284	-118.8304
607	CSM Unit 3	363029.3212	5310369.0399	47.9320	-118.8338
606	CSM Unit 3	363791.7652	5311551.4066	47.9428	-118.8240
606	CSM Unit 3	362853.6428	5311396.1753	47.9412	-118.8365
606	CSM Unit 3	363565.6871	5310783.2682	47.9358	-118.8268
606	CSM Unit 3	362719.5815	5310811.8786	47.9359	-118.8381
605	CSM Unit 3	361977.7758	5312566.5373	47.9515	-118.8486
605	CSM Unit 3	362738.2816	5312556.8751	47.9516	-118.8384
604	CSM Unit 3	360767.7616	5312910.7008	47.9544	-118.8649
604	CSM Unit 3	361535.2357	5312529.4221	47.9511	-118.8545
603	CSM Unit 3	359502.9597	5312933.1314	47.9543	-118.8818
602	CSM Unit 3	357524.0949	5312862.1105	47.9532	-118.9083
601	CSM Unit 3	356539.8783	5311225.6969	47.9383	-118.9209
599	CSM Unit 3	354493.9201	5311585.8752	47.9411	-118.9484
598	CSM Unit 3	352811.0635	5312741.3546	47.9511	-118.9713

**Notes:**

CSM = Conceptual Site Model

A Cultural Resources Working Group review of the proposed sample locations convened on August 7, 2012 and approved sediment sampling within 150 feet (Area = 70,686 ft<sup>2</sup> = 1.6 acres) of each of the above-listed sediment sampling positions (Letter from Dr. Laura Buelow of the U.S. Environmental Protection Agency dated August 24, 2012).

Table B1-2. Summary of Bioassay Endpoints

28-d <i>Hyalella azteca</i>		10-d <i>Chironomus dilutus</i>		42-d <i>Hyalella azteca</i>		Long-term <i>Chironomus dilutus</i>	
• Survival	• Weight & biomass	• Survival	• Weight & biomass	• Survival (at Days 28, 35, and 42)	• Weight & biomass (at Days 28 and 42)	• Survival (at Day 20)	• Weight and biomass (at Day 20)
						• Male and female emergence	
				• Reproduction (at Days 35 and 42)		• Adult mortality	
				• Number of adult males (at Days 42)		• Number of egg cases oviposited	
				• Number of adult females (at Days 42)		• Number of eggs produced	
						• Number of hatched eggs	

**Note:**

Survival is measured as the number of surviving organisms divided by the initial number of organisms.

Weight is measured as the dry weight (*H. azteca*) or ash-free dry weight (*C. dilutus*) of surviving organisms divided by the number of surviving organisms.

Biomass is measured as the dry weight (*H. azteca*) or ash-free dry weight (*C. dilutus*) of surviving organisms divided by the initial number of organisms.

Reproduction is measured as the number of young divided by the number of females.

Table B1-3. Test Conditions for Conducting a 28-d Sediment Toxicity Test with *Hyalella azteca*

Parameter	Conditions
Test type	Whole-sediment toxicity test with renewal of overlying water
Temperature	23 ± 1°C
Light quality	Wide-spectrum fluorescent lights
Illuminance	About 500 lux
Photoperiod	16L:8D
Test chamber	300-mL high-form lipless beaker
Sediment volume	100 mL
Overlying water volume	175 mL
Renewal of overlying water	2 volume additions/d; continuous or intermittent (e.g., 1 volume addition every 12 h)
Age of organisms <sup>a</sup>	7- to 8-d old at the start of the test with a goal of achieving starting weights in the range of 0.02 to 0.035 mg/organism. The weight of a representative sample of organisms at the start of sediment exposures will be documented.
Number of organisms/chamber	10
Number of replicate chambers/treatment <sup>a</sup>	14 replicates: 8 for biological endpoints and 6 for chemistry only
Feeding <sup>a</sup>	YCT food: fed 1.0 mg YCT/day to each test chamber during Days 0 to 13, and 2 mg YCT/day to each test chamber during the remaining exposure (Days 14 to 27).
Aeration	None, unless DO in overlying water drops below 2.5 mg/L.
Overlying water <sup>a</sup>	Test water will consist of reconstituted water created using the methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide.
Test chamber cleaning	If screens become clogged during a test, gently brush the <i>outside</i> of the screen.
Overlying water quality	Hardness, alkalinity, conductivity, pH, and ammonia at the beginning and end of a test. Temperature daily. Conductivity weekly. DO and pH three times/week. Concentrations of DO should be measured more often if DO drops more than 1 mg/L since the previous measurement.
Test duration <sup>a</sup>	28 d
Endpoints	Survival, weight, and biomass
Test acceptability	Minimum mean control survival of 80% on Day 28.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

DO = dissolved oxygen

Table B1-4. Recommended Test Conditions for Conducting a 10-d Sediment Toxicity Test with *Chironomus dilutus*

Parameter	Conditions
Test type	Whole-sediment toxicity test with renewal of overlying water
Temperature	23 ± 1°C
Light quality	Wide-spectrum fluorescent lights
Illuminance	About 500 lux
Photoperiod	16L:8D
Test chamber	300-mL high-form lipless beaker
Sediment volume	100 mL
Overlying water volume	175 mL
Renewal of overlying water	2 volume additions/d; continuous or intermittent (e.g., 1 volume addition every 12 h)
Age of organisms <sup>a</sup>	Second- to third-instar larvae (about 10-d-old larvae; all organisms must be third instar or younger with approximately 50% of the organisms at second instar and approximately 50% of the organisms at third instar; goal to achieve a starting average weight of 0.12 mg/organism). The weight of a representative sample of organisms at the start of sediment exposures will be documented.
Number of organisms/chamber	10
Number of replicate chambers/treatment <sup>a</sup>	11 replicates: 8 for biological endpoints and 3 for chemistry only
Feeding <sup>a</sup>	TetraMin® goldfish food, 6 mg of particles fed daily to each test chamber.
Aeration	None, unless DO in overlying water drops below 2.5 mg/L.
Overlying water <sup>a</sup>	Reformulated moderately hard reconstituted water (as specified in USEPA [2000] page 25)
Test chamber cleaning	If screens become clogged during a test, gently brush the <i>outside</i> of the screen.
Overlying water quality	Hardness, alkalinity, conductivity, pH, and ammonia at the beginning and end of a test. Temperature and DO daily.
Test duration	10 d
Endpoints	Survival, weight, and biomass (AFDW)
Test acceptability	Minimum mean control survival must be 70%, with minimum mean weight/surviving control organism of 0.48 mg AFDW.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

AFDW = ash-free dry weight

DO = dissolved oxygen

Table B1-5. Test Conditions for Conducting a 42-d Sediment Toxicity Test with *Hyalella azteca*

Parameter	Conditions
Test type	Whole-sediment toxicity test with renewal of overlying water
Temperature	23 ± 1°C
Light quality	Wide-spectrum fluorescent lights
Illuminance	About 500 lux
Photoperiod	16L:8D
Test chamber	300-mL high-form lipless beaker
Sediment volume	100 mL
Overlying water volume	175 mL in the sediment exposure from Day 0 to Day 28 (175 to 275 mL in the water-only exposure from Day 28 to Day 42)
Renewal of overlying water	2 volume additions/d; continuous or intermittent (e.g., 1 volume addition every 12 h)
Age of organisms <sup>a</sup>	7- to 8-d-old at the start of the test with a goal of achieving starting weights in the range of 0.02 to 0.035 mg/organism. The weight of a representative sample of organisms at the start of sediment exposures will be documented.
Number of organisms/chamber	10
Number of replicate chambers/treatment <sup>a</sup>	18 replicates: 12 for biological endpoints and 6 for chemistry only. Of the 12 replicates for biological endpoints, 4 replicates are for 28-d survival and growth and 8 replicates are for 35- and 42-d survival, growth, and reproduction.
Feeding <sup>a</sup>	YCT food: fed 1.0 mg YCT/day to each test chamber during Days 0 to 13, and 2 mg YCT/day to each test chamber during the remaining exposure (Days 14 to 42).
Aeration	None, unless DO in overlying water drops below 2.5 mg/L.
Overlying water <sup>a</sup>	Test water will consist of reconstituted water created using the methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide.
Test chamber cleaning	If screens become clogged during a test, gently brush the <i>outside</i> of the screen.
Overlying water quality	Hardness, alkalinity, conductivity, and ammonia at the beginning and end of a sediment exposure (Day 0 and 28). Temperature daily. Conductivity weekly. DO and pH three times/week. Concentrations of DO should be measured more often if DO drops more than 1 mg/L since the previous measurement.
Test duration	42 d
Endpoints	28-d survival, weight, and biomass; 35-d survival and reproduction; and 42-d survival, weight, biomass reproduction, and number of adult males and females on Day 42.
Test acceptability	Minimum mean control survival of 80% on Day 28.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

DO = dissolved oxygen

Table B1-6. Test Conditions for Conducting a Long-term Sediment Toxicity Test with *Chironomus dilutus*

Parameter	Conditions
Test type	Whole-sediment toxicity test with renewal of overlying water
Temperature	23 ± 1°C
Light quality	Wide-spectrum fluorescent lights
Illuminance	About 500 lux
Photoperiod	16L:8D
Test chamber	300-mL high-form lipless beaker
Sediment volume	100 mL
Overlying water volume	175 mL
Renewal of overlying water	2 volume additions/d; continuous or intermittent (e.g., 1 volume addition every 12 h)
Age of organisms	< 24-h-old larvae. The weight of a representative sample of organisms at the start of sediment exposures will be documented.
Number of organisms/chamber	12
Number of replicate chambers/treatment <sup>a</sup>	25 replicates: 16 for biological endpoints and 9 for chemistry only. Of the 16 replicates for biological endpoints, 4 replicates are created only to produce auxiliary males.
Feeding <sup>a</sup>	TetraMin® goldfish food, 6 mg of particles fed daily to each test chamber starting Day 1
Aeration	None, unless DO in overlying water drops below 2.5 mg/L.
Overlying water <sup>a</sup>	Reformulated moderately hard reconstituted water (as specified in USEPA 2000 page 25)
Test chamber cleaning	If screens become clogged during a test, gently brush the <i>outside</i> of the screen.
Overlying water quality	Hardness, alkalinity, conductivity, and ammonia at the beginning, on Day 20, and at the end of a test. Temperature daily (ideally continuously). DO and pH three times/week. Conductivity weekly. Concentrations of DO should be measured more often if DO has declined by more than 1 mg/L since the previous measurement.
Test duration	About 50 to 65 d; each treatment is ended separately when no additional emergence has been recorded for seven consecutive days. When no emergence is recorded from a treatment, termination of that treatment should be based on the control sediment using this 7-d criterion.
Endpoints	20-d survival, weight, and biomass; female and male emergence, adult mortality, the number of egg cases oviposited, the number of eggs produced, and the number of hatched eggs.
Test acceptability	Average size of <i>C. dilutus</i> in the control sediment at 20 d must be at least 0.6 mg/surviving organism as dry weight or 0.48 mg/surviving organism as AFDW. Emergence should be greater than or equal to 50%. Experience has shown that pupae survival is typically >83% and adult survival is >96%. Time to death after emergence is <6.5 d for males and <5.1 d for females. The mean number of eggs/egg case should be greater than or equal to 800 and the percent hatch should be greater than or equal to 80%.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

AFDW = ash-free dry weight

Table B1-7. Test Acceptability Requirements for a 28-d Sediment Toxicity Test with *Hyalella azteca*

A. It is recommended for conducting a 28-d test with <i>Hyalella azteca</i> that the following performance criteria be met	
1.	Age of <i>H. azteca</i> at the start of the test should be 7- to 8-d old with a goal of achieving starting weights in the range of 0.02 to 0.035 mg/organism <sup>a</sup> . Starting a test with substantially younger or older organisms may compromise the reproductive endpoint.
2.	Average survival of <i>H. azteca</i> in the control sediment on Day 28 should be greater than or equal to 80%. Mean weight of <i>H. azteca</i> in the control sediment on Day 28 should be greater than or equal to 0.4 mg dry/individual. <sup>a</sup>
3.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the sediment exposure, and DO should be maintained above 2.5 mg/L in the overlying water.
B. Performance-based criteria for culturing <i>H. azteca</i> include the following	
1.	It may be desirable for laboratories to periodically perform 96-h water-only reference-toxicity tests to assess the sensitivity of culture organisms. Data from these reference-toxicity tests could be used to assess genetic strain or life-stage sensitivity of test organisms to select chemicals.
2.	Laboratories should track parental survival in the cultures and record this information using control charts if known-age cultures are maintained. Records should also be kept on the frequency of restarting cultures and the age of brood organisms.
3.	Laboratories should record the following water quality characteristics of the cultures at least quarterly: pH, hardness, alkalinity, and ammonia. Dissolved oxygen in the cultures should be measured weekly. Temperature of the cultures should be recorded daily. If static cultures are used, it may be desirable to measure water quality more frequently.
4.	Laboratories should characterize and monitor background contamination and nutrient quality of food if problems are observed in culturing or testing organisms.
5.	Physiological measurements such as lipid content might provide useful information regarding the health of the cultures.
C. Additional requirements	
1.	All organisms in a test must be from the same source. If organisms are purchased, vendor information must be reported.
2.	All test chambers (and compartments) should be identical and should contain the same amount of sediment and overlying water.
3.	Standard negative-control sediment, quartz sand negative control sediment <sup>a</sup> , and appropriate solvent controls must be included in a test. The concentration of solvent used must not adversely affect test organisms.
4.	Test organisms must be cultured and tested at 23°C (±1°C).
5.	The daily mean test temperature must be within ±1°C of 23°C. The instantaneous temperature must always be within ±3°C of 23°C.
6.	Natural physio-chemical characteristics of test sediment collected from the field should be within the tolerance limits of the test organisms (see USEPA [2000] for standard tolerance limits).
7.	Source of overlying water and control sediments must be documented and reported.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letter from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

DO = dissolved oxygen



Table B1-8. Test Acceptability Requirements for a 10-d Sediment Toxicity Test with *Chironomus dilutus*

A.	It is recommended for conducting a 10-d test with <i>C. dilutus</i> that the following performance criteria be met
1.	Tests must be started with second- to third-instar larvae (about 10-d-old larvae) with a goal of achieving a starting average weight of 0.12 mg/organism <sup>a</sup> .
2.	Average survival of <i>C. dilutus</i> in the control sediment must be greater than or equal to 70% at the end of the test.
3.	Average size of <i>C. dilutus</i> in the control sediment must be at least 0.48 mg AFDW at the end of the test.
4.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the test, and DO should be maintained above 2.5 mg/L in the overlying water.
B.	Performance-based criteria for culturing <i>C. dilutus</i> include the following
1.	It may be desirable for laboratories to periodically perform 96-h water-only reference-toxicity tests to assess the sensitivity of culture organisms. Data from these reference-toxicity tests could be used to assess genetic strain or life-stage sensitivity of test organisms to select chemicals.
2.	Laboratories should keep a record of time to first emergence for each culture and record this information using control charts. Records should also be kept on the frequency of restarting cultures.
3.	Laboratories should record the following water quality characteristics of the cultures at least quarterly: pH, hardness, alkalinity, and ammonia. DO in the cultures should be measured weekly. Temperature of the cultures should be recorded daily. If static cultures are used, it may be desirable to measure water quality more frequently.
4.	Laboratories should characterize and monitor background contamination and nutrient quality of food if problems are observed in culturing or testing organisms.
5.	Physiological measurements such as lipid content might provide useful information regarding the health of the cultures.
C.	Additional requirements
1.	All organisms in a test must be from the same source. If organisms are purchased, vendor information must be reported.
2.	All test chambers (and compartments) should be identical and should contain the same amount of sediment and overlying water.
3.	Standard negative-control sediment, quartz sand negative control sediment <sup>a</sup> , and appropriate solvent controls must be included in a test. The concentration of solvent used must not adversely affect test organisms.
4.	Test organisms must be cultured and tested at 23°C (±1°C).
5.	The daily mean test temperature must be within ±1°C of 23°C. The instantaneous temperature must always be within ±3°C of 23°C.
6.	Natural physio-chemical characteristics of test sediment collected from the field should be within the tolerance limits of the test organisms. (see USEPA [2000] for standard tolerance limits).
7.	Source of overlying water and control sediments must be documented and reported.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

AFDW = ash-free dry weight

DO = dissolved oxygen

Table B1-9. Test Acceptability Requirements for a 42-d Sediment Toxicity Test with *Hyalella azteca*

<b>A.</b> It is recommended for conducting a 42-d test with <i>H. azteca</i> that the following performance criteria be met	
1.	Age of <i>H. azteca</i> at the start of the test should be 7- to 8-d-old with a goal of achieving starting weights in the range of 0.02 to 0.035 mg/organism <sup>a</sup> . Starting a test with substantially younger or older organisms may compromise the reproductive endpoint.
2.	Average survival of <i>H. azteca</i> in the control sediment on Day 28 should be greater than or equal to 80%. Mean weight of <i>H. azteca</i> in the control sediment should be greater than or equal to 0.4 mg dry/individual on Day 28, and greater than or equal to 0.5 mg dry/individual on Day 42. <sup>a</sup>
3.	Laboratories participating in round-robin testing reported after 28-d sediment exposures in a control sediment, survival >80% for >88% of the laboratories; length >3.2 mm/individual for >71% of the laboratories; and dry weight >0.15 mg/individual for >66% of the laboratories. Reproduction from Day 28 to Day 42 was >2 young/female for >71% of the laboratories participating in the round-robin testing. Reproduction was more variable within and among laboratories; hence, more replicates might be needed to establish statistical differences among treatments with this endpoint.
4.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the sediment exposure, and DO should be maintained above 2.5 mg/L in the overlying water.
<b>B.</b> Performance-based criteria for culturing <i>H. azteca</i> include the following	
1.	It may be desirable for laboratories to periodically perform 96-h water-only reference-toxicity tests to assess the sensitivity of culture organisms. Data from these reference-toxicity tests could be used to assess genetic strain or life-stage sensitivity of test organisms to select chemicals.
2.	Laboratories should track parental survival in the cultures and record this information using control charts if known-age cultures are maintained. Records should also be kept on the frequency of restarting cultures and the age of brood organisms.
3.	Laboratories should record the following water quality characteristics of the cultures at least quarterly: pH, hardness, alkalinity, and ammonia. DO in the cultures should be measured weekly. Temperature of the cultures should be recorded daily. If static cultures are used, it may be desirable to measure water quality more frequently.
4.	Laboratories should characterize and monitor background contamination and nutrient quality of food if problems are observed in culturing or testing organisms.
5.	Physiological measurements such as lipid content might provide useful information regarding the health of the cultures.
<b>C.</b> Additional requirements	
1.	All organisms in a test must be from the same source. If organisms are purchased, vendor information must be reported.
2.	All test chambers (and compartments) should be identical and should contain the same amount of sediment and overlying water.
3.	Standard negative-control sediment, quartz sand negative control sediment <sup>a</sup> , and appropriate solvent controls must be included in a test. The concentration of solvent used must not adversely affect test organisms.
4.	Test organisms must be cultured and tested at 23°C (±1°C).
5.	The daily mean test temperature must be within ±1°C of 23°C. The instantaneous temperature must always be within ±3°C of 23°C.
6.	Natural physio-chemical characteristics of test sediment collected from the field should be within the tolerance limits of the test organisms. (See USEPA [2000] for standard tolerance limits).
7.	Source of overlying water and control sediments must be documented and reported.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

DO = dissolved oxygen

Table B1-10. Test Acceptability Requirements for a Long-term Sediment Toxicity Test with *Chironomus dilutus*

A. It is recommended for conducting a long-term test with <i>C. dilutus</i> that the following performance criteria be met	
1.	Tests must be started with less than 1-d- (<24-h) old larvae. Starting a test with substantially older organisms may compromise the emergence and reproductive endpoint.
2.	Average survival of <i>C. dilutus</i> in the control sediment must be greater than or equal to 70% on Day 20 and greater than 65% at the end of the test.
3.	Average size of <i>C. dilutus</i> in the control sediment at 20 d must be at least 0.6 mg/surviving organism as dry weight or 0.48 mg/surviving organism as AFDW. Emergence should be greater than or equal to 50%. Experience has shown that pupae survival is typically >83% and adult survival is >96%. Time to death after emergence is <6.5 d for males and <5.1 d for females. The mean number of eggs/egg case should be greater than or equal to 800 and the percent hatch should be greater than or equal to 80%.
4.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the test, and DO should be maintained above 2.5 mg/L in the overlying water.
B. Performance-based criteria for culturing <i>C. dilutus</i> include the following	
1.	It may be desirable for laboratories to periodically perform 96-h water-only reference-toxicity tests to assess the sensitivity of culture organisms. Data from these reference-toxicity tests could be used to assess genetic strain or life-stage sensitivity of test organisms to select chemicals.
2.	Laboratories should keep a record of time to first emergence for each culture and record this information using control charts. Records should also be kept on the frequency of restarting cultures.
3.	Laboratories should record the following water quality characteristics of the cultures at least quarterly: pH, hardness, alkalinity, and ammonia. DO in the cultures should be measured weekly. Temperature of the cultures should be recorded daily. If static cultures are used, it may be desirable to measure water quality more frequently.
4.	Laboratories should characterize and monitor background contamination and nutrient quality of food if problems are observed in culturing or testing organisms.
5.	Physiological measurements such as lipid content might provide useful information regarding the health of the cultures.
C. Additional requirements	
1.	All organisms in a test must be from the same source. If organisms are purchased, the vendor information must be reported.
2.	All test chambers (and compartments) should be identical and should contain the same amount of sediment and overlying water.
3.	Standard negative-control sediment, quartz sand negative control sediment <sup>a</sup> , and appropriate solvent controls must be included in a test. The concentration of solvent used must not adversely affect test organisms.
4.	Test organisms must be cultured and tested at 23°C (±1°C).
5.	The daily mean test temperature must be within ±1°C of 23°C. The instantaneous temperature must always be within ±3°C of 23°C.
6.	Natural physio-chemical characteristics of test sediment collected from the field should be within the tolerance limits of the test organisms. (See USEPA [2000] for standard tolerance limits).
7.	Source of overlying water and control sediments must be documented and reported.

Source: USEPA (2000)

**Notes:**

<sup>a</sup> Modified from EPA standard method as directed by EPA (letters from Shawn D. Blocker on June 21, 2012 and Dr. Laura Buelow on August 24, 2012)

AFDW = ash-free dry weight

DO = dissolved oxygen

Table B3-1. Sampling Containers, Preservation, and Holding Time Requirements for Sediment Chemistry

Priority	Analysis	Container		Filtered	Preservation	Holding Time	Proposed Laboratory		Total Minimum Sample Size Needed <sup>a, b</sup>
		Type	Size				Sample Size	Size	
1	TAL metals, percent moisture								
	EPA 6020A metals					6 months	10 g		
	EPA 6010C metals	WMG	8 oz	NA	4±2°C	6 months	10 g		
2	Mercury					28 days	5 g		
2	pH					7 days	20 g		
2	Total organic carbon					28 days	1 g		337 g
2	AVS/SEM	WMG	8 oz	NA	No headspace, 4±2°C	14 days	25 g		
3	Grain size	WMG	4 oz	NA	4±2°C	6 months	100 g		
3	Backscatter electron microscopy	WMG	16 oz	NA	4±2°C	NA	5 g		
3	Archival						161 g		
1	Bioassay	Plastic	15 gal	NA	4±2°C	ASAP	12 gal		12 gal

**Notes:**

<sup>a</sup> Total sample size does not include additional sample volumes needed for laboratory quality control or field duplicate samples. If sufficient sample volume is available, attempt to fill all sample containers provided. If insufficient sample volume is available, fill containers to laboratory minimums in order of priority and then fill the priority containers with any remaining sample.

<sup>b</sup> Project field duplicate samples should be collected for 10 percent of all analytical samples and submitted blind to the analytical laboratory. In addition, EPA split samples (containing at least 200 g) will be collected for 15 percent of all analytical samples by EPA.

ASAP = as soon as possible

AVS/SEM = acid volatile sulfide/simultaneously extracted metals

TAL = target analyte list

NA = not applicable

WMG = wide-mouth glass

Table B3-2. Sample Containers, Preservation, and Holding Time Requirements for Porewater

Priority Rating		Container		Preservation	Holding Time	Proposed Minimum Laboratory Sample Size (mL) <sup>a</sup>
		Type	Size (mL)			
<b>1</b>	<b>Dissolved TAL Metals</b>					
	Aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, nickel, selenium, silver, thallium, vanadium, and zinc	HDPE	50	1 mL of 20% HNO <sub>3</sub> , pH<2, 4±2°C	6 months	20
	Calcium, iron, magnesium, potassium, and sodium					20
<b>2</b>	<b>Organic Carbon</b>					
	DOC <sup>b</sup>	HDPE	50	4 ± 2°C	28 days	25
	TOC				28 days	20
<b>3</b>	<b>Conventional Parameters <sup>c</sup></b>					
3.1	pH				ASAP	15
3.2	Alkalinity as CaCO <sub>3</sub>				14 days	25
3.3	Hardness <sup>d</sup>	HDPE	50	4 ± 2°C	6 months	25
3.4	Chloride, sulfate				28 days	10
Syringe volume = maximum porewater volume available (mL) =						135
						140

**Notes:**

<sup>a</sup> If sufficient sample volume is available, attempt to fill all sample containers provided. If insufficient sample volume is available, fill containers to laboratory minimums in order of priority and then fill the priority containers with any remaining sample.

<sup>b</sup> The chain-of-custody for DOC must be marked "lab filter needed"

<sup>c</sup> Minimum sample volumes for the hierarchy of listed conventional analytes to be analyzed by the laboratory are as follows: pH = 15 mL; alkalinity/hardness = 25 mL; and

<sup>d</sup> Hardness will be calculated per: equivalent CaCO<sub>3</sub> = 2.5 (mg Ca<sup>2+</sup>/L) + 4.1(mg Mg<sup>2+</sup>/L).

ASAP = as soon as possible

CaCO<sub>3</sub> = calcium carbonate

DOC = dissolved organic carbon

HDPE = high density polyethylene bottle

HNO<sub>3</sub> = nitric acid

TAL = target analyte list

TOC = total organic carbon

Table B4-1. Number of Samples for Analytical Chemistry and Bioassay Measurements

Sample Analysis <sup>a</sup>	Media	Number of Sediment Sampling Locations	Number of Replicate Chambers per Location	Number of Assessments per Chamber	Number of Analyses from all Locations
<b>Analytical Chemistry</b>					
Sediment	Sediment	140	NA	NA	140
Field porewater	Porewater	140	NA	NA	140
<b>Bioassay</b>					
28-d <i>H. azteca</i>	Biota	74	8	1	592
Lab porewater	Porewater (peeper) <sup>b</sup>	74	6	0.3	148
	Porewater (centrifuge) <sup>c</sup>	74	NA	NA	74
10-d <i>C. dilutus</i>	Biota	74	8	1	592
Lab porewater	Porewater (peeper) <sup>b</sup>	74	3	0.3	74
	Porewater (centrifuge) <sup>c</sup>	74	NA	NA	74
42-d <i>H. azteca</i> <sup>d</sup>	Biota	18	12	1	216
Lab porewater	Porewater (peeper) <sup>b</sup>	18	6	0.3	36
	Porewater (centrifuge)	18	NA	NA	18
Long-term <i>C. dilutus</i> <sup>d</sup>	Biota	18	16 <sup>e</sup>	1	288 <sup>e</sup>
Lab porewater	Porewater (peeper) <sup>b</sup>	18	9	0.3	54
	Porewater (centrifuge)	18	NA	NA	18
				Sediment total	140
				Biota total	1,688
				Porewater total	636

**Notes:**

<sup>a</sup> Does not include routine water quality monitoring (e.g., for temperature, dissolved oxygen, pH, conductivity) that will be conducted to ensure that the tests are conducted under standard conditions.

<sup>b</sup> Lab porewater will be collected using Brumbaugh type peepers in the "chemistry only" replicate test chambers. Porewater from the Brumbaugh type peepers in three replicate test chambers will be combined into a single sample to provide as much volume as possible for analytical measurements.

<sup>c</sup> Lab porewater will be sampled from each sediment sample selected for short-term toxicity tests at the start of exposures using centrifugation. These porewater samples will be analyzed for DOC, pH, alkalinity, sulfide, major cations, and major anions to inform the BLM for interpreting toxicity data.

<sup>d</sup> Bulk sediment chemistry, porewater metals (from peepers), and BLM parameters (from centrifuged sediment) will be analyzed anew prior to longer-term reproduction toxicity tests.

<sup>e</sup> Four test chambers will be populated with *C. dilutus* for each sediment location in order to produce auxiliary males for possible use in the latter steps of the long-term *C. dilutus* bioassay tests. These chambers are not true replicates and will not be measured for biological endpoints.

NA = not applicable

Table B5-1. Measurement Quality Objectives for Sediment Samples

Parameter	Analytical Accuracy (% recovery)	Analytical Precision (relative % deviation)	Overall Completeness (percent)
Metals	75-125	20	90
Mercury	75-125	20	90
TOC	70-125	20	90
AVS	55-145	45	90
SEM	75-125	30	90
Grain Size	NA	NA	NA

**Notes:**

AVS = acid volatile sulfide

SEM = simultaneously extracted metals

TOC = total organic carbon

NA = not applicable

Table B5-2. Measurement Quality Objectives for Porewater Samples

Analysis	Analytical Accuracy (% recovery)	Analytical Precision (relative % deviation)	Overall Completeness (percent)
TOC and DOC	80-120	17	90
Alkalinity as CaCO <sub>3</sub>	NA	20	90
Hardness as CaCO <sub>3</sub>	90-120	20	90
Cations/anions	90-110	20	90
Metals and metalloids	75-125	20	90

**Notes:**

CaCO<sub>3</sub> = calcium carbonate

TOC = total organic carbon

DOC = dissolved organic carbon

NA = not applicable